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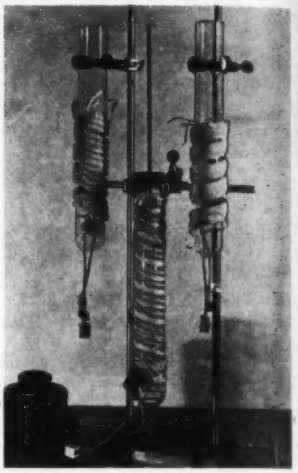

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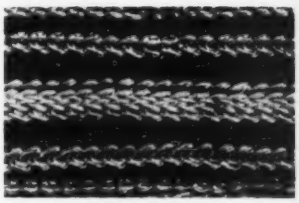

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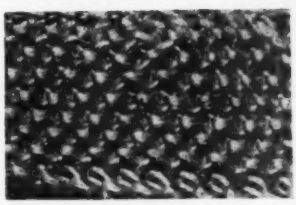

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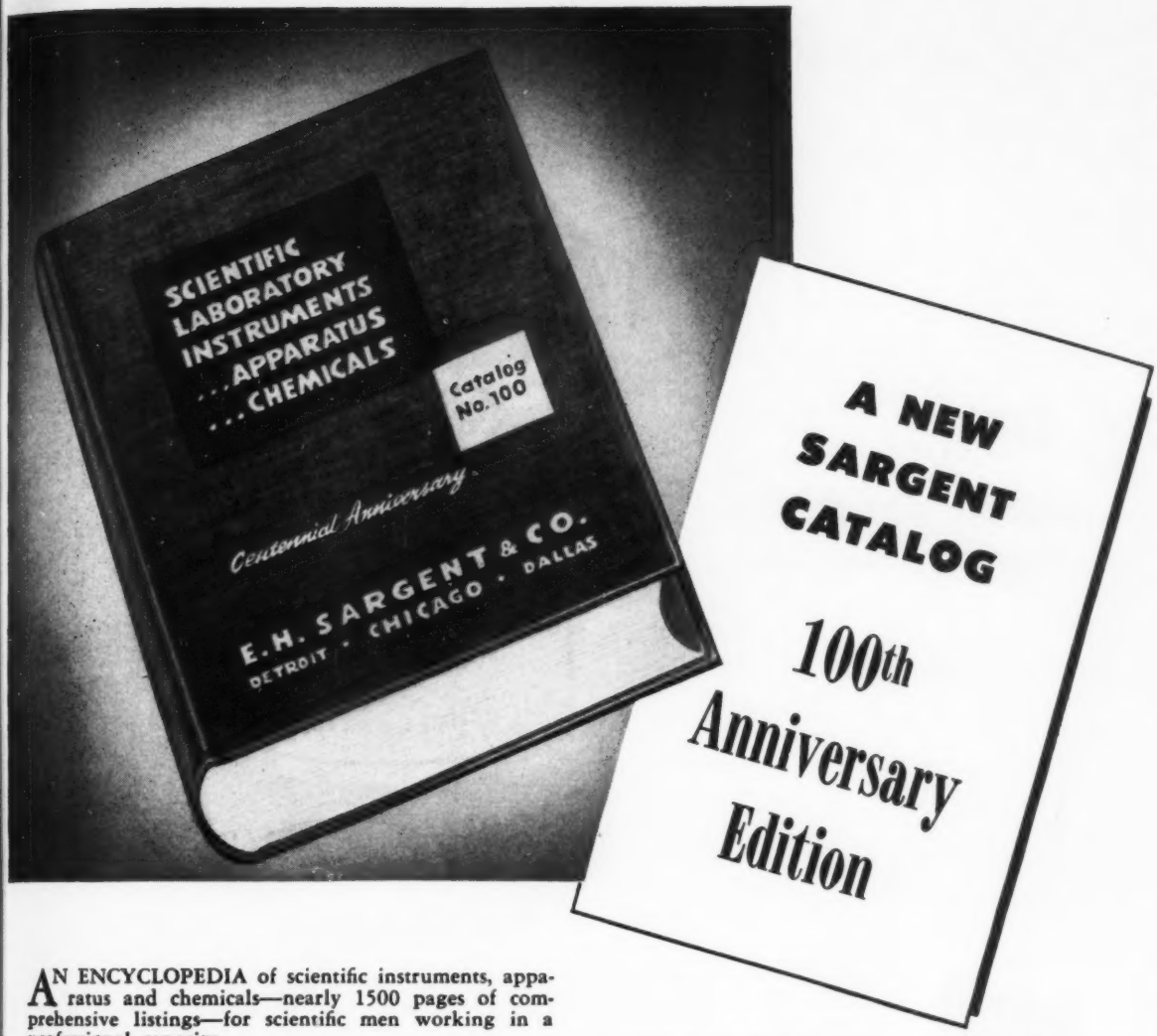

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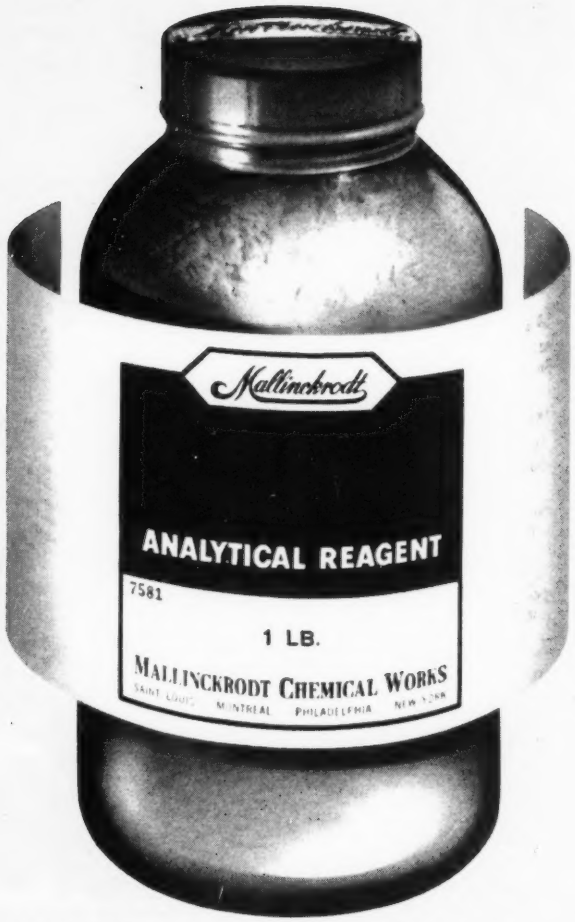

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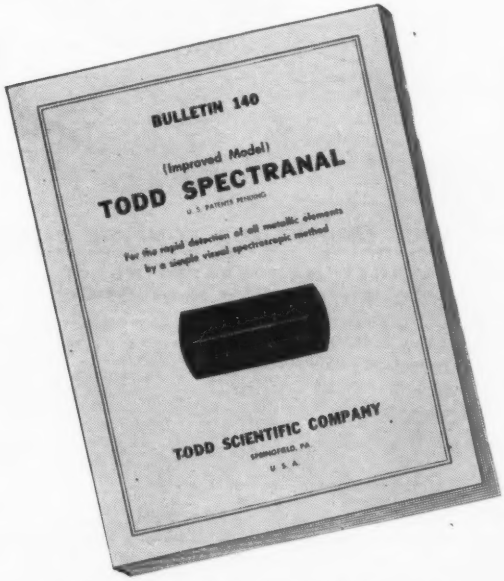
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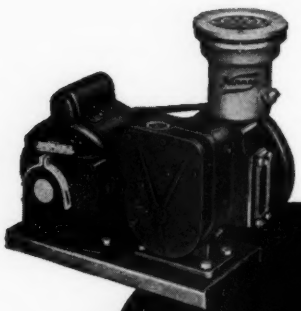

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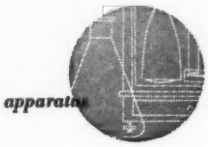

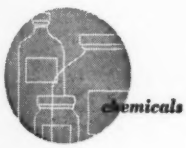

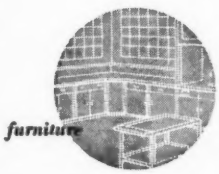

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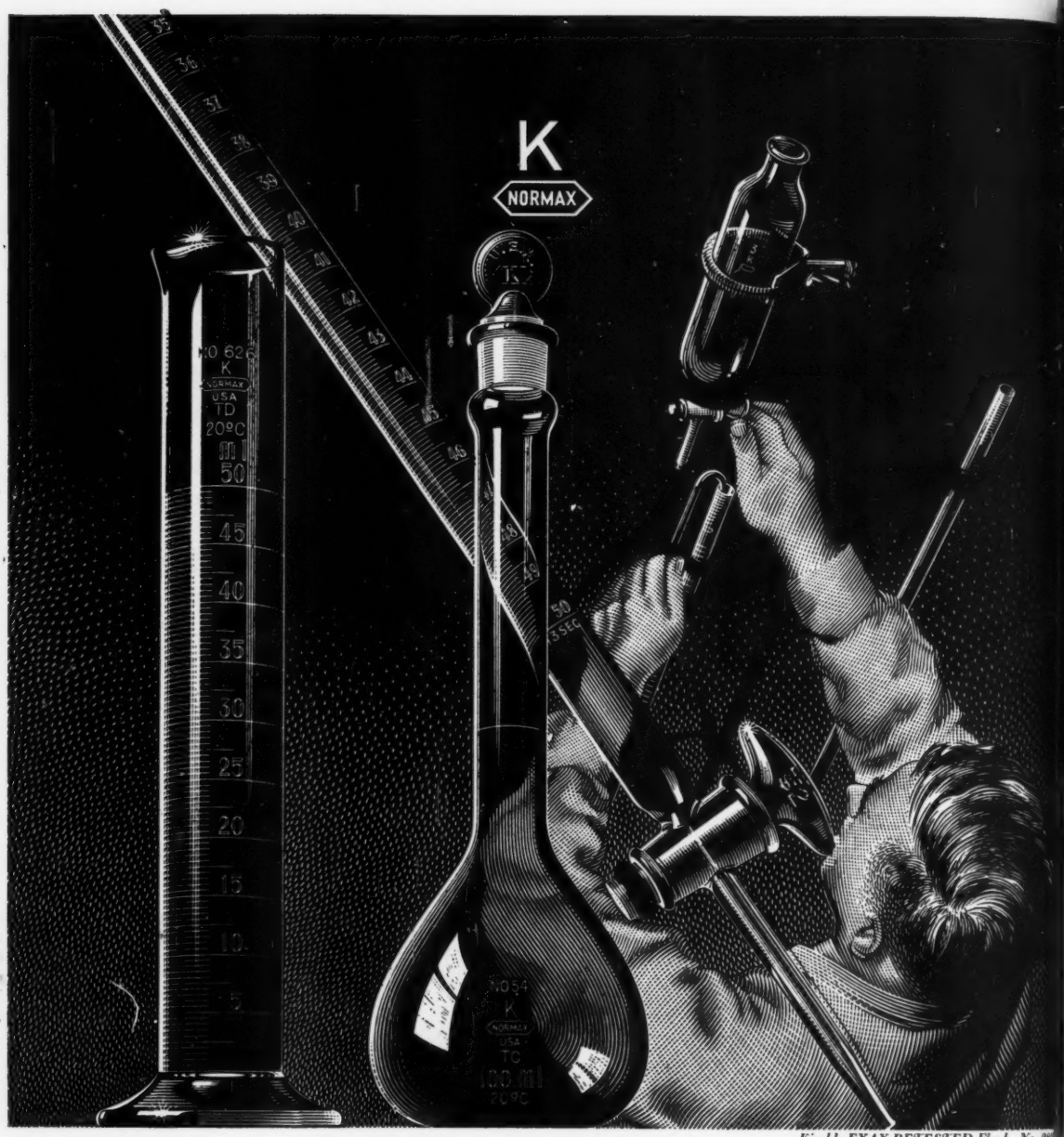

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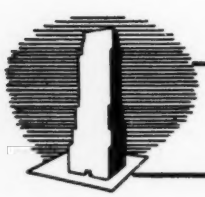

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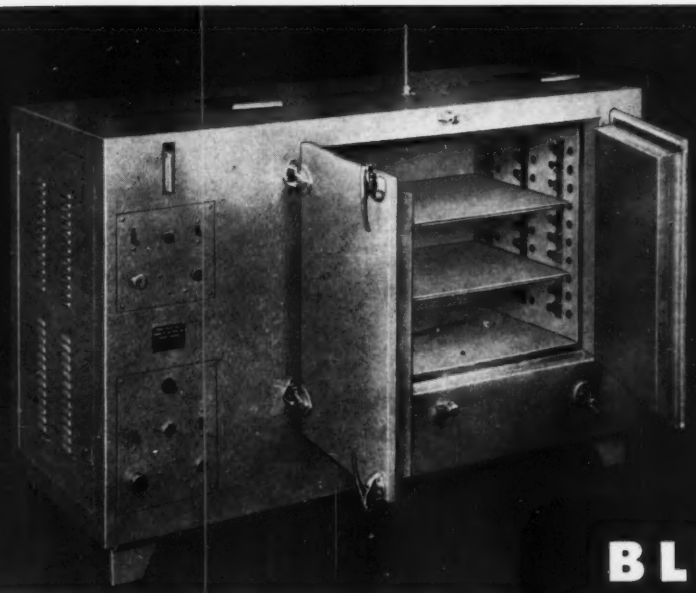

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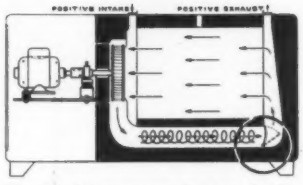

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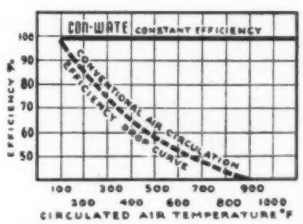

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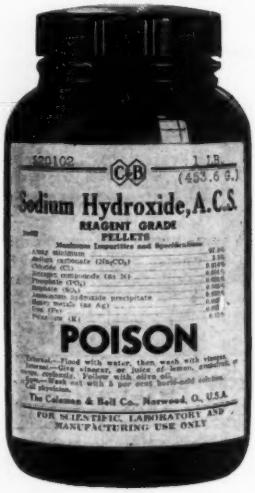

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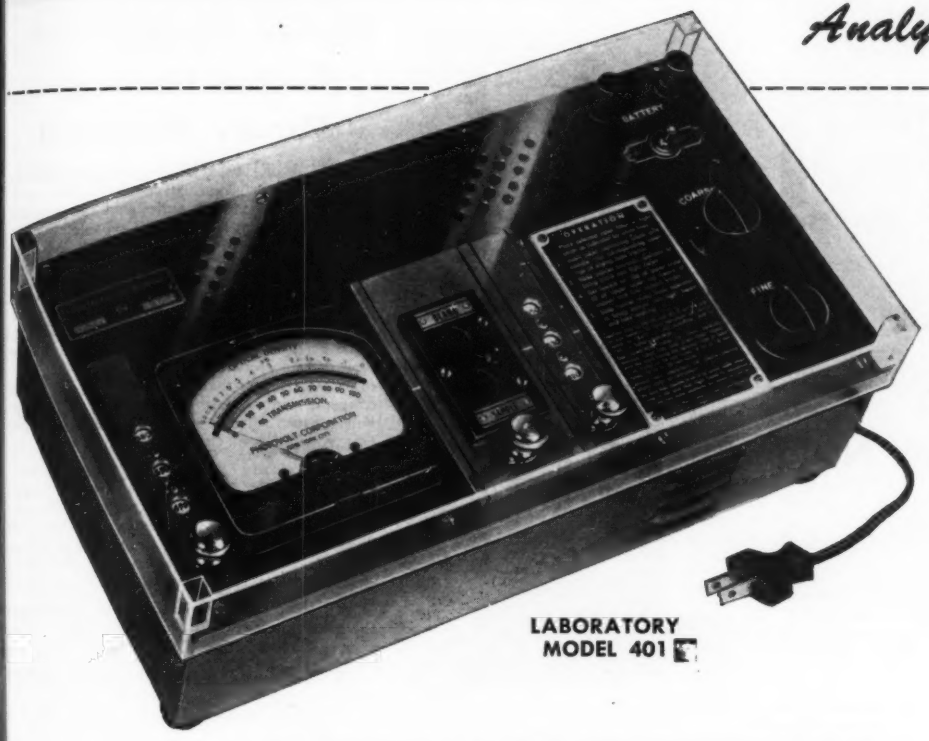

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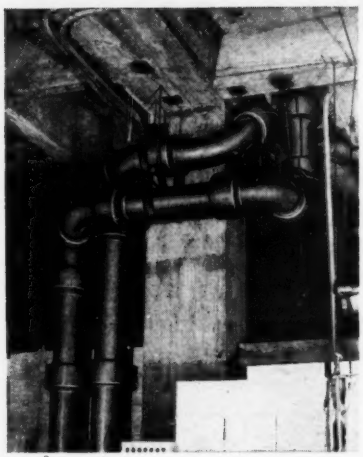

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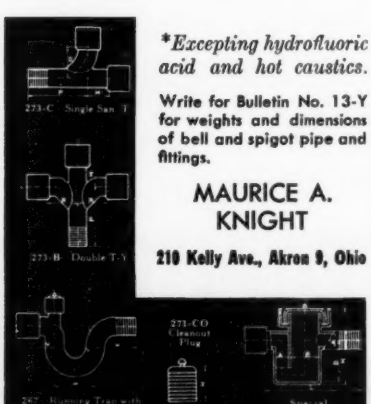

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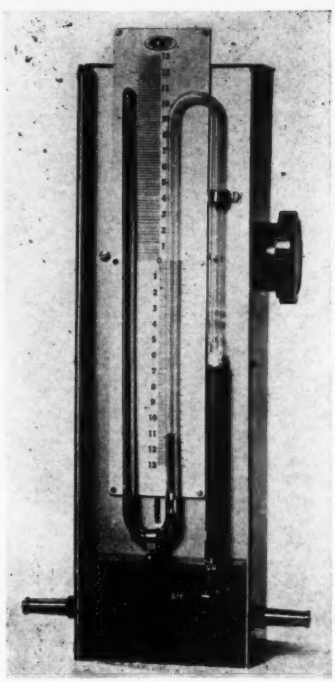

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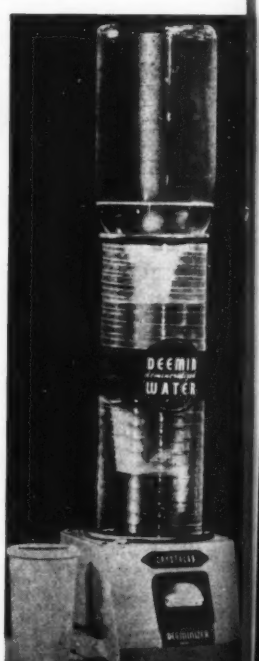

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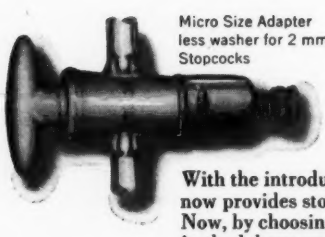

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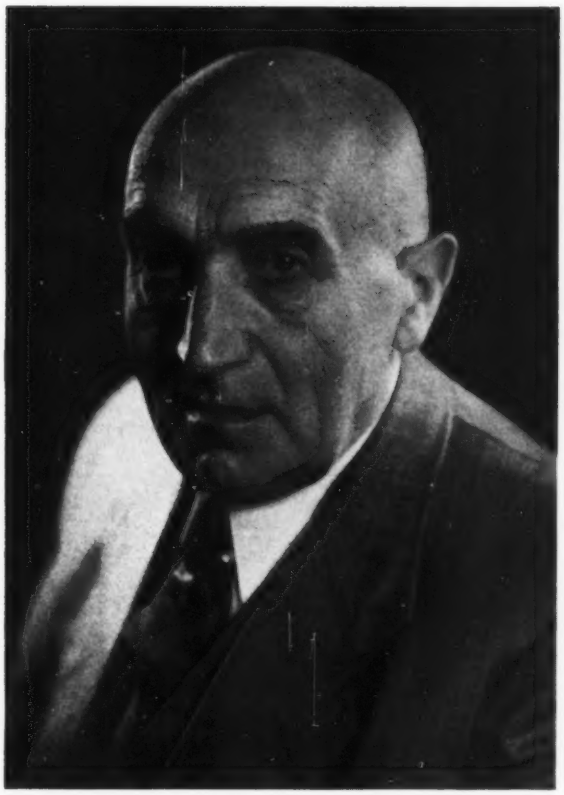

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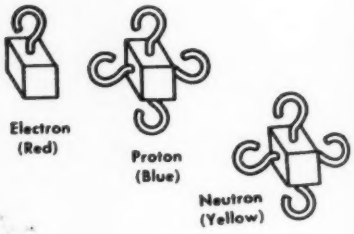

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Editor's Outlook

SOME remarks published recently in our contemporary, *Chemical Age*, show that Britain has many of the same problems we have, not the least of which is that of recruiting good science teachers at the prevailing low salaries and in competition with the more remunerative opportunities in industry. The following might almost as well have been written to describe the situation in our country.

Recently in one of the better-class Sunday newspapers a well-known academician suggested that industrial leaders would be better drawn from the students of arts and humanistic subjects than from the science. Others want to see the curricula for science widened by the inclusion of broader subjects, e.g., some of the so-called arts. Only occasionally is it pointed out that education begins in schools and that the main preoccupation of headmasters and governing bodies today is to find anyone who is prepared to teach scientific subjects for the low remuneration offered; it would be almost amusing if it were not so tragic to listen to so many voices extolling this or that recipe for the ideal university education and drowning the many fewer voices that point to this truly serious crack in the foundation—the dwindling reserves of science teachers in the schools and the inadequate flow of new recruits.

The academician who believes that arts students will cope better with industry's management problems will certainly see his theory put to the test; and those who want a much broadened training for scientists can certainly expect a considerable leavening of pre-university education. These things will happen whether anybody plans them or not. Indeed, in a generation or so it may be possible for those who now distrust the more specialised scientific educations to pat themselves on the back by proving that industry is steadily increasing the proportion of arts scholars in its intake of recruits. Industry, however, will have no alternative. Less and less first-class material will survive the diminishing capacities of schools to teach science, especially chemistry, well; and what does survive will be avidly seized by the universities and groomed for lifetime service in specialised science. Year by year the standard of science-teaching in our schools drops back, simply because the payment for teaching science is much below the standard of payment for practising science. For every word that is said about this, a hundred are spoken about reconstructing university methods and introducing new curricula and objectives. Industry's future needs are primarily shaped in schools; universities complete and refine these processes but they cannot create the basic patterns.

[1] *Chemical Age*, Dec. 15, 1951, p. 787.

Erratum: Several readers called attention to the careless mistake in August's Outlook. Even the editor knows that $\frac{d}{dx}(x^2)$ should be $2x$!

INSTRUCTIONAL ERROR IN QUANTITATIVE ANALYSIS

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For many years the authors have felt that there is much wrong with some of the common determinations as taught in elementary quantitative analysis. Perhaps the greatest single observation giving rise to this feeling was that good students on the whole did not achieve significantly better accuracy on such determinations than average or even poor students. At first this observation did not appear significant, for there were many ways of "explaining" it or of looking at student results in ways such as to make this observation appear erroneous. Recently, however, it has become apparent that such inferior determinations are almost valueless for teaching the student constructively or for rating him accurately. Over the past few years data have been accumulated which not only prove and substantiate this belief, but which also indicate that the number of such poor determinations may be far greater than originally suspected.

In this paper the suggestion is advanced that in such determinations the error which a student makes is influenced by errors beyond his control. Error beyond control of the student is primarily the responsibility of the instructor, and is termed instructional error. The detrimental consequences of such instructional error are discussed, and criteria are given for its revelation. The pervasive and insidious nature of instructional error is demonstrated, and it is emphasized that critical examination of student results is necessary before an instructor may assure himself of the absence of such error.

The above beliefs regarding instructional error arose and were supported by a rather long and critical study of the determination of chloride as silver chloride. After elimination of several sources of error (all of which were instructional), good students achieved relative errors within 0.1 per cent of the truth and $^5/_6$ of the students in the class achieved relative errors within 0.3 per cent. This is an experimental refutation of the belief that beginning students should not be expected to work at such error levels for the silver chloride experiment. A detailed description of this study is given later in this paper.

It is hoped that this work will not be interpreted by the reader as a pointless confession of errors rampant and endemic at the University of Wisconsin. On the contrary, it is believed that instructional error might well be pandemic. The single and paramount purpose in publishing this paper is the hope that the reader will apply some of the suggested criteria to an examination of his own experiments, if he is a teacher of quantitative analysis.

THE NATURE OF INSTRUCTIONAL ERROR

Factors Affecting the Accuracy of the Student's Analysis. The *over-all error* of a student's result, which is the only error measurable by the instructor, may be contributed to by three sources: (1) the *error inherent in the method* of analysis used, (2) the *student's error*, and (3) *instructional error*. Each of these classes requires further definition.

The error inherent in the method of analysis used may be defined as that made by an experienced analyst working with good equipment and facilities of the type used by the student. For the tested methods used in quantitative analysis, this error is usually stated in the directions, or may be inferred from them. (For many of the common determinations, this error is small, of the order of 0.1 per cent or less.) This error is random in nature, and any student falling within this error should be classed as excellent. Further, there can be no differentiation among students in this range of error, for the magnitude of the error made is a matter of chance.

The student's error, as contrasted to his over-all error, is that which is under his control. Contributions to this error come from inadequacy of technique or judgment, or from failure to apply properly principles or directions in which instruction has been given. It is this student's error which is the measure of his ability, and not necessarily his over-all error, as is shown in the following paragraphs.

All error not inherent in the method and not due to the student is classed as instructional error. Sources of such error are faulty equipment, facilities, chemicals, or unknowns, and erroneous or inadequate directions.

Now may be stated a prime requisite of a good experiment in quantitative analysis, in so far as instructional purposes are concerned. This is that the student's result must not be affected appreciably by instructional error, or in other words, that instructional error must be negligible. The over-all error of a student's result can be no measure of his ability unless instructional error is small. If instructional error is predominant, the accuracy and precision with which a student works is beyond his control. Whether or not the instructional error influences a particular student's result may be a matter of chance, as may be the extent of the influence.

When instructional error is predominant, a good student may return a result classed as "poor" as often as "good"; the poor student may do likewise, and the student's over-all error is a meaningless quantity.

Consequences of Appreciable Instructional Error. It is necessary that the student be correctly apprised of his error on each analysis that he performs, so that he may re-examine, reinterpret, and improve his technique and knowledge in the light of that error. Such an examination of error is an integral and most important part of all experimental work, not only in quantitative analysis, but also in other fields of science. When the student's error is obscured by the instructor's, the student is deprived of this opportunity, and all that quantitative analysis can teach him is to perform unquestioningly what the instructor has decreed are the proper motions of the good technician. If the student is informed that his analysis is poor, all he can decide is to go uncritically through the same motions again and to repeat the experiment in its entirety; if he is informed that the result is passing, but mediocre, he must accept this, for he has no assurance of better results if he repeats the work. In short, the presence of instructional error teaches resignation and apathy, with little opportunity to develop the sense of constructive criticism and inquiry so necessary to every good scientific worker.

It should be emphasized that the reason for concern over instructional error and the correct grading of experiments is not primarily to give the student a fair grade. The reason for concern lies far deeper than this. A grade on an experiment rendered unreliable because of instructional error prevents the student from ever knowing how well he has done on the experiment. Without this knowledge he simply cannot work intelligently.

Indications of the Existence of Instructional Error. There are some symptoms which are reliable indications of the existence of instructional error. Categorically, these are:

(1) An almost certain sign of the presence of instructional error is given when the errors obtained by the students in the determination are poorly correlated with the ratings of the students on any other related, valid basis. When many students who are known by other standards to be good frequently do poorly on the determination, or when many average or poor students achieve errors comparable to the good students, instructional error is probably the cause.

(2) Instructional error is probably present if most of the good students cannot achieve the error inherent in the method of analysis used. (This is a relative error of 0.1 per cent for many of the basic determinations.)

(3) Instructional error is probably predominant when there is poor correlation between the student's precision (as measured by the average deviation or range of his determinations) and his accuracy (as measured by his over-all error, or difference between his reported value and the true value known by the instructor).

These symptoms are related to certain long-standing controversies among teachers of analytical chemistry. For instance, regarding the first symptom, it is believed by some instructors that there are "many" students who are good in theory but poor in laboratory work (and vice versa). Regarding the second symptom, some instructors believe it both undesirable and unfeasible that students be required to work at the same error level as an accomplished analyst for a good rating. Regarding the third symptom, some instructors believe that little significance can be attached to the precision of student work, even if no grading weight is attached to precision and no inducement is offered to the student to "fudge" his results into high precision.

In other words, many instructors will question the validity of these indications of instructional error on first reading them. In the limited space available, it is not possible to "justify" these criteria, nor to counter the popular (and often erroneous!) arguments that are made against them. The ultimate justification and acceptance of these criteria must come from the mind of the reader.

In general support of these criteria, and to help the reader gain acceptance of them, it must be stated that all the above symptoms are observed to diminish or disappear as instructional errors are reduced or eliminated. (See the following study of the silver chloride determination.)

The Reduction of Instructional Error. By the preceding symptoms it is possible to discover the presence of instructional error. When discovered in any particular experiment the question then arises not only as to how such error may be reduced but also how far it should be reduced.

For an experiment which has not been rigorously checked for several years there may be several specific sources of instructional error, and discovery and reduction of these may be very difficult. In general it is of assistance to plot the distribution of errors and precision for all students in a class. These plots often allow a decision as to the nature of the errors (*i. e.*, whether predominantly determinate or random, and the sign and constancy of the error if determinate), and thereby aid in localizing the source.

But the methods for discovering each specific error and the means of reducing it cannot be generalized for these vary with each particular experiment at hand.

Usually it is neither practical nor possible to reduce instructional error much below that inherent in the method of analysis used. Instructional error may be regarded as sufficiently reduced when the symptoms given in the preceding section of this paper disappear on critical examination. It should be cautioned that instructional error remains hidden to superficial examination of student results. Thus, attention to selected results may prove very misleading. Also, the fact that a "good portion" of the class works within 0.1–0.2 per cent of the truth is not proof at all of the absence of instructional error, as is shown later in this paper.

DETERMINATION OF CHLORIDE AS SILVER CHLORIDE

Student Data Over Three Semesters. In Figure 1*a* is plotted the error of each student who performed the gravimetric determination of chloride, for all elementary classes of Fall, 1950. Thorne Smith samples containing 25–50 per cent of chloride were analyzed according to established directions then in use. Each point on the plot in Figure 1*a* represents a student. The horizontal line at 0 per cent relative error represents the true value for each student, and the vertical distance of each point from the horizontal line represents the relative error(in per cent) of the average of three determinations performed by the student on his sample. Only students reporting three determinations are shown in Figure 1. (Points falling at the margins at 1.5 and −1.5 per cent errors indicate errors at or beyond these values, falling off the diagram.) Superficial examination of Figure 1*a* might lead to the conclusion that this experiment is a passable one. The median student error is +0.42 per cent. (This is only 0.1–0.2 absolute per cent of chloride, since the samples contained only 25–50 per cent of chloride.) Also, over 13 per cent of the students are within 0.1 per cent of the truth, and half of the class is within 0.54 per cent of the truth. However, these figures alone mean little, and allow no safe conclusions as to the presence or absence of instructional error.

Further information is contained in Figure 2*a*, which is a scatter diagram of the precision versus the accuracy of each student in the class shown in Figure 1*a* (Fall, 1950). Each point in Figure 2*a* represents the error

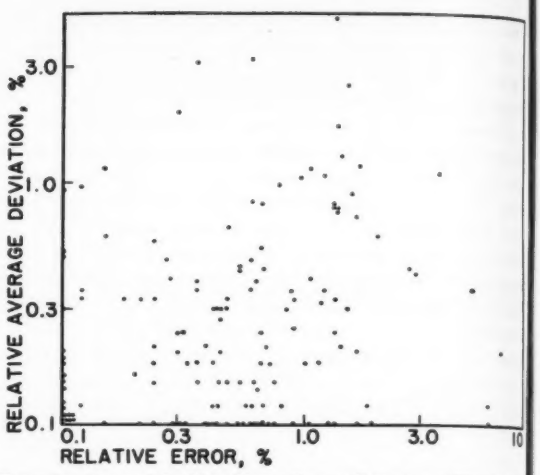

Figure 2a. Error of the Mean Versus Average Deviation of Three Determinations (Class of Fall, 1950)

(abscissae) of the average and the average deviation (ordinates) of the three determinations performed by each student. (To keep this logarithmic plot within reasonable bounds, errors and average deviations below 0.1 per cent are not considered significantly different from 0.1 per cent. Whenever a student achieves less than 0.1 per cent error or average deviation, his point is located on the 0.1 per cent error ordinate or the 0.1 per cent average deviation abscissa, respectively.)

It may be seen from Figure 2*a* that there is virtually no correlation between precision and accuracy. High-precision students work with about the same accuracy as all students except perhaps those with very low precision. Also, not apparent from Figure 2*a*, since student grades are not listed thereon, good, average, and poor students all have about the same median average deviation. These observations all indicate the predominance of instructional error.

In Spring, 1951, attention was focused on a small class, still large enough for significant conclusions, and the following changes were made: (1) New drying ovens were installed with sufficient capacity, operating in the range 110–130°C. for drying samples, and in the range 140–160°C. for drying silver chloride. (2) Balances were overhauled. (3) Thorne Smith samples containing 25–30 per cent chloride were used. (4) Directions

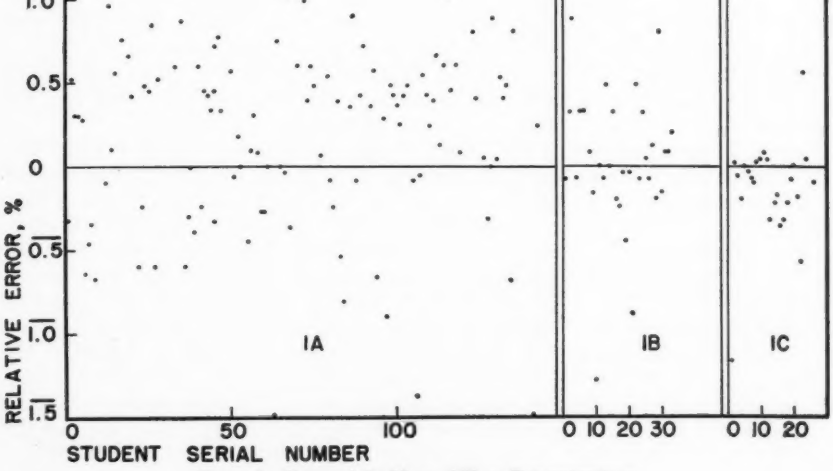

Figure 1. Errors of the Mean of Three Determinations

a. Class of Fall, 1950
b. Class of Spring, 1951
c. Class of Summer, 1951

analysis in the text by Pierce and Haenisch[1] were used.

The errors of the 34 students in this class are plotted in Figure 1*b*. There is unquestionably an improvement over the class of Fall, 1950. The median error is only +0.02 per cent, indicating virtually no bias on the student results. Also, 38 per cent of the class is within 0.1 per cent of the truth, and $^5/_6$ of the class is within 0.4 per cent of the truth.

No attempt was made to resolve the instructional errors which were eliminated. The differences between Figures 1*a* and 1*b* were considered sufficiently explained by supposing that several instructional errors which were variable and different in sign might have been reduced by the changes made.

Figure 2*b* is a scatter diagram of the accuracy and precision of the students in the class of Figure 1*b*.

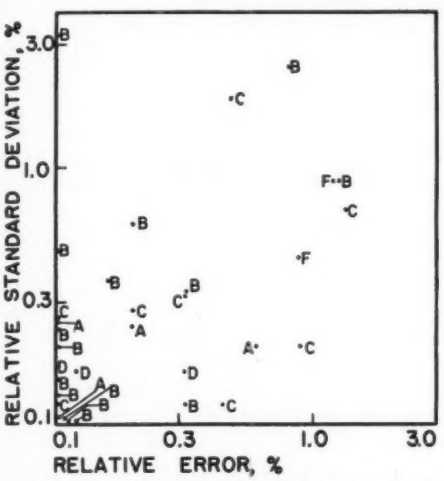

Figure 2b. Error of the Mean Versus Standard Deviation of Three Determinations (Class of Spring, 1951)

Definite correlation appears between the accuracy of a student's result and the precision with which he works, in that there seems to be a trend of points from the lower left to the upper right regions of the diagram. Students with passable accuracy (within 0.5 per cent of the truth) work with considerably higher precision on the average than those with poor accuracy (error greater than 0.5 per cent).

Yet the correlation in Figure 2*b* is not good. Students falling within 0.1 per cent of the truth do not work with significantly better or worse precision than those lying between 0.1–0.2 per cent or 0.2–0.5 per cent of the truth. It seems that this experiment is not capable of distinguishing very well between students in adjacent grade categories, but only between passing and poor students. That is, the instructor may say from a student's accuracy whether the student has done passably or poorly, but nothing more than this. Apparently, instructional error still exists.

[1] Pierce, W. C., and E. L. Haenisch, "Quantitative Analysis," 3rd. ed., John Wiley & Sons, Inc., New York, 1948, pp. 381–3.

The suspicion of instructional error is strengthened when the final grade received by each student in the course is placed beside his point in Figure 2*b*, or when the cross-classification chart in Table 1 is constructed. These final grades are probably a fairly reliable rating of the general ability of the student, being based on quiz grades, laboratory performance, and the personal opinion of the instructors. While there is some correlation between the accuracy at which a student works and his final grade, the correlation is not good. An appreciable proportion of good and average students achieve no better accuracy or precision on this determination than the poor students, and an appreciable proportion of the poor and average students obtain about the same accuracy or precision as the good ones. There is still some error-causing factor, operating beyond the control of the student, which obscures the accuracy with which he works. This experiment is still quite valueless for instruction and for rating the student, for the instructor cannot say with reasonable assurance that a student who obtains inaccurate results has really performed poorly.

It was suspected that perhaps the sulfate used as the diluent in the unknown was causing a positive but nonreproducible error, due to coprecipitation. In the class of 26 students in Summer, 1951, the two following changes were made: (1) The silver chloride was digested overnight instead of the 1–2 hours called for originally (ref. 1); (2) Sulfate-free unknowns containing 48–50 per cent chloride were prepared by mixing reagent grade primary standard potassium chloride with small amounts of sodium chloride.

The errors of students in this class of Summer, 1951, are plotted in Figure 1*c*. The median error of the class is −0.08 per cent, a considerably greater bias than for the preceding class of Spring, 1951. However, it should be noted that over 50 per cent of the students fall within 0.1 per cent of the truth, and that $^5/_6$ of the class is within 0.3 per cent of the truth. Examinations of the scatter diagram in Figure 2*c* confirms the belief that the

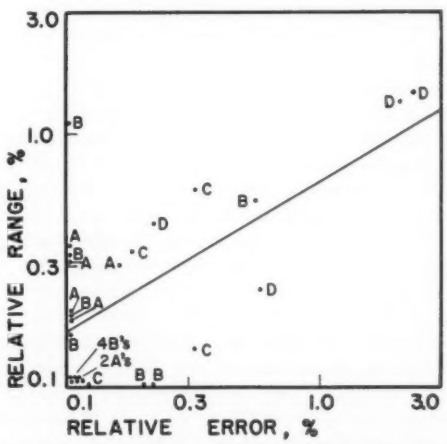

Figure 2c. Error of the Mean Versus Range of Three Determinations (Class of Summer, 1951)

experiment has been improved, for there is somewhat better correlation of precision with accuracy than in the case of Figure 2*b*. Further, the final course grades, shown for each student beside his point in Figure 2*c*, show much better correlation with the accuracy with which the student works than in the case of Figure 2*b*. The correlation between final course grades and accuracy achieved on the experiment is more clearly shown in Table 2.

The conclusion is that the determination as performed in the summer of 1951 is a fairly good one, since most symptoms of instructional error have disappeared. This is not true for the determination as performed in the Spring of 1951 (Figures 1*b*, 2*b*).

An interpretation of the difference between the classes of Summer, 1951, and Spring, 1951, is as follows: In the class of Summer, 1951, there is operative only one potentially predominant source of error: loss of silver chloride due to faulty technique. This error is entirely within the control of the student, for he is instructed how to avoid losses and to obtain quantitative recovery. On the whole, the good students avoid this error and the poor ones do not, and the error of a student's result is a measure of his ability. Since poor students are definitely present in the class, they bias the class median, making it low. In the class of Spring, 1951, this error due to loss of silver chloride is probably compensated and obscured by the instructional error of coprecipitation. This is an instructional error because it is beyond control of the student. Not only is this error an unknown one, but the directions do not avoid it. While it is always a positive error, the extent to which it affects each student's result is a matter of chance. Such an error, if significant, can affect a good student's results only adversely, while it may affect the poor student's result beneficially as well as adversely. In short, this instructional error has a leveling effect and irons out the distinction between good and poor students, which is in accord with the observations of the class of Spring, 1951. In the presence of this error, the excellence of a student's technique may not be well correlated with the accuracy of the result.

TABLE 1
Cross Classification Chart for Error Versus Final Course Grade (Class of Spring, 1951)

Relative error, %	*Final course grades*				
	A	*B*	*C*	*D*	*F*
0.1, or below	2	2	2	1	...
Over 0.1, to 0.2	1	4	1	1	...
Over 0.2, to 0.4	...	2	1	1	...
Over 0.4, to 0.8	1	1	2	...	2
Over 0.8	...	...	...	...	...

The nature of the error has only been postulated for purposes of the above discussion. In a later section of this paper data are given which support this postulate and indicate the magnitude of the error, as well as the factors upon which it depends.

Grading of the Gravimetric Determination of Chloride in the Absence of Instructional Error. Inspection of Figure 2*c* shows the median range of the best students (those with errors less than 0.1 per cent) is about 0.18 per cent. From this range, the standard deviation of a single determination of the median best student may be estimated[2] as 0.11 per cent for three determinations. Hence, the relative standard error of the average of the three determinations of the median best student is $0.11/\sqrt{3} = 0.06$ per cent.

It would be spurious to grade on the basis of an interval any less than 0.06 per cent, which is a measure of the minimum random error, unavoidable even by the best students except for chance. Reference to the normal error curve shows that a good student has 60 per cent as good a chance of making an error of 0.06 per cent as he does of achieving the truth. There is no significant difference between a 0.06 per cent error and no error.

Thus, to grade on a schedule such as one which deducts one point from a perfect grade of 100 per 0.01 per cent difference from the truth assigns spurious differences to students. There can be no such fine distinction, even among excellent students, and for students with larger errors (*i. e.* two or three times the standard error of 0.06 per cent), this system breaks down altogether. Such an arithmetical scheme for grading would require that a student with 0.2 per cent error receive an 80, while one with 0.4 per cent error receive a 60, and one with 0.6 per cent error receive a 40. There is simply not this much difference among these students. If the grading is made on the basis of one point per 0.02 per cent error or more, then the difficulty in the lower grade categories is reduced; but the difficulty in the higher grade categories is aggravated, for too many students who do not deserve to be are classed as excellent.

Instead, the following geometric grading schedule is suggested:

Error	*Grade*
0.1% or below	10 (Excellent)
over 0.1 to 0.2%	9
over 0.2 to 0.4%	8
over 0.4 to 0.8%	7
over 0.8 to 1.5%	6
over 1.5%	5

A grade of 5 points is a minimum grade, given for faithful performance of the experiment, but indicating very poor accuracy. Of course, for experiments with inherently poorer accuracy, the intervals may be broadened. The specific schedule given above is suggested only for the gravimetric determination of silver chloride when instructional error is absent, and for other experiments with inherent errors of the order of 0.1 per cent or less.

On this basis, there is little difference between the student with 0.5 per cent error and one with 1 per cent error. This seems logical, for both have performed poorly and made large errors; the fact that the error of

[2] DIXON, W. J., AND F. J. MASSEY, "Introduction to Statistical Analysis," McGraw-Hill Book Co., Inc., New York, 1951, p. 329.

one student is only half that of the other is probably a matter of chance and should be assigned little significance. The same reasoning assigns more reasonable differences than the arithmetic scale to the students making 0.2, 0.4, and 0.6 per cent errors.

For stricter grading, to reduce the number of students in the higher grade intervals, 0.06 per cent could have been adopted as the grading interval instead of 0.1 per cent. However, it should be noted from Figure 2*c* and Table 2 that correlation between accuracy and precision disappears below 0.1–0.2 per cent error, as does that between final grades and accuracy. Therefore, instructional error may still be operative at this level. For safety's sake, it is considered better to use the wider interval recommended above. It is believed that any results within 0.1 per cent of the truth should be regarded as excellent for the elementary analytical unknown sample by comparison with the "average value" or "most probable value" obtained by the class on that unknown may be even more faulty. In any case, this procedure gives no check on instructional error whatsoever.

Error Due to Sulfate. To verify the assumption that sulfate in the unknowns caused a positive error, samples of potassium chloride weighed as primary standard were analyzed under conditions similar to those in the text by Pierce and Haenisch (ref. 1). The yields obtained were compared with the theoretical yield calculated from the amounts of potassium chloride in the samples. All experiments were performed in duplicate. Results are summarized in Table 3.

Experiments 1 through 4 were carried out almost as directed in the text by Pierce and Haenisch, which specifies an amount of silver nitrate precipitant 10 per

TABLE 3
Error Due to Sulphate and Excess Silver Nitrate in Gravimetric Determination of Chloride

	Yield of AgCl obtained				$AgNO_3$ precipitant				
Experiment	Theoretical, g.	Experimental, g.	Error, mg.	Error, %	Mols. Na_2SO_4 per mol. of chloride	Excess used over theoretical required, %	Concentration, % $AgNO_3$	Volume, ml.	Digestion time, hrs.
1a	0.5022	0.5019	−0.3	−0.06	0	40	2	200	18
b	0.5022	0.5019	−0.3	−0.06					
2a	1.0044	1.0037	−0.7	−0.07	0	40	2	200	18
b	1.0044	1.0036	−0.8	−0.08					
3a	1.0044	1.0039	−0.5	−0.05	0	40	10	120	18
b	1.0044	1 0039	−0.5	−0.05					
4a	1.0044	1.0044	0.0	0.00	0	40	10	120	1
b	1.0044	1.0043	−0.1	−0.01					
5a	0.5018	0.5022	0.4	0.08	1.0	350	10	200	1
b	0.5018	0.5021	0.3	0.06					
6a	0.2509	0.2509	0.0	0.00	1.0	350	10	200	1
b	0.2509	0.2518	0.9	0.36					
7a	0.2509	0.2513	0.4	0.16	2.0	650	10	200	1
b	0.2509	0.2520	1.1	0.44					
c	0.2509	0.2517	0.8	0.32					
d	0.2509	0.2518	0.9	0.36					
8a	0.2509	0.2510	0.1	0.04	2.0	650	10	200	24–48
b	0.2509	0.2511	0.2	0.08					
c	0.2509	0.2512	0.3	0.12					
d	0.2509	0.2514	0.5	0.20					
9a	0.5018	0.5021	0.3	0.06	0	350	10	200	18
b	0.5018	0.5021	0.3	0.06					
10a	0.5018	0.5023	0.5	0.10	1.0	60	2	200	18
b	0.5018	0.5021	0.3	0.06					

course, and that any differences within this range should be regarded as insignificant.

It should be noted here that the practice of grading student results by comparison with values obtained by the instructor "under similar conditions of analysis" is a particularly treacherous one, for instructional error may flourish undetected under such circumstances. If the instructor, on using the student method for analyzing a particular unknown, obtains a value significantly different from the value obtained from another reliable method or source, then grading on the basis of either value is meaningless. The only solution to the difficulty is to eliminate the discrepancy, or to obtain sufficient assurance as to the nature and reproducibility of the error so that it may be properly corrected.

The practice of grading student results on a particular

cent in excess of that which would be required, assuming the sample to be pure sodium chloride. On digesting overnight, yields are definitely but insignificantly low, probably due to mechanical losses, or to coprecipitation of chloride as sodium chloride. On digesting for only the hour specified in the text, the yield is very close to theoretical. The precision is very high in all four experiments.

Experiments 5 through 8 were performed in the presence of potassium sulfate, to simulate analyses on potassium chloride-potassium sulfate mixtures. The amount of silver nitrate precipitant used was calculated by assuming the whole sample to be pure sodium chloride, and was greatly in excess of the theoretical amount required. The yields in these experiments differ significantly in two respects from the yields in experiments

1 through 4, where no sulfate was present: (1) While in the absence of sulfate, experimental yields are very slightly below the theoretical, yields in the presence of sulfate are higher than theoretical by as much as 0.4 per cent in some cases. (2) Precision is much poorer in the presence of sulfate. An explanation which accounts for these differences is that silver sulfate is coprecipitated rather non-reproducibly under the conditions of experiments 5 through 8.

It is conceivable that students with moderately large amounts of sulfate in their samples might approach the conditions of experiments 5 through 8, and might make uncontrolled positive errors of the order of several tenths of a percent. The non-reproducible errors of experiments 5 through 8, which are as high as 0.4 per cent should be regarded as minimum errors, since these were obtained by an accomplished analyst. The errors obtained by the student due to this source could well be even greater than those in Table 3. It seems, therefore, that the data in Table 3 provide an adequate explanation of the errors made by students in the class of Spring, 1951, as shown in Figures 1*b*, 2*b*, and Table 1.

To find whether the errors in experiments 5 to 8 were due to the large excess of silver nitrate used, or to the presence of sulfate alone, experiments 9 and 10 were performed. It appears that rather large excesses of silver nitrate or the presence of moderate amounts of sulfate each cause slight positive errors which are not serious; these errors become serious only when both factors operate simultaneously, and when the digestion time is short.

Conclusions. There are several interesting conclusions which may be drawn from this experimental study, all of which support the categorical statements of the preceding section.

(1) The presence of instructional error may be well concealed from superficial examination of the accuracy of student analyses. For instance, the fact that a "reasonable" proportion of the class obtains results within 0.1–0.2 per cent of the truth is no criterion at all of the absence of instructional error. This is so because student results are usually spread sufficiently so that an appreciable proportion of the class may be found within 0.1–0.2 per cent of any value over quite a range which may be regarded as the truth. Likewise, the fact that the class median error is small is no indication of the absence of instructional error, for this may be acting randomly and predominantly, either alone, or to balance other errors due either to the instructor or the student.

(2) It is interesting to note that every source of error which was removed to increase the accuracy of the student's work was the instructor's responsibility, and that this error operated beyond the student's control and predominated over his error. Further, these errors were found to exist, destroying validity of any judgment on the student's ability, in an experiment regarded before investigation as one of the better experiments of the elementary course in quantitative analysis.

(3) When instructional error is removed, good students obtain results within 0.1 per cent of the truth, and most of the class falls within errors only a few fold greater than this. This is a direct refutation of the argument that most students are incapable of achieving relative errors less than 0.1–0.2 per cent. If they cannot do so, it may be the instructor's fault rather than the students'. It should be emphasized that after removal of instructional error there is no choice but to teach this determination on the level of a 0.1 per cent relative error, and to demand this of the good student.

TABLE 2
Cross Classification Chart for Error Versus Final Course Grade (Class of Summer, 1951)

Relative error, %	*Final course grades*				
	A	*B*	*C*	*D*	*F*
0.1, or below	1	3	...	...	...
Over 0.1, to 0.2	1	1	2	...	...
Over 0.2, to 0.4	...	1	2	1	...
Over 0.4, to 0.8	...	1	...	1	...
Over 0.8, to 1.5	...	...	...	...	...
Over 1.5	...	...	...	...	2

(4) Safe criteria of the absence of instructional error are those given in the first section of this paper, namely: (*a*) Correlation between the accuracy achieved by the student on the experiment and his rating by another reliable method. (*b*) Achievement by most of the good students of errors around that inherent in the method of analysis used. (*c*) Correlation between the student's accuracy and precision.

(5) It appears more reasonable to grade students according to their errors on a geometric scale rather than on the frequently used arithmetic scale.

ACKNOWLEDGMENT

The authors wish to thank their colleagues for giving so generously of their time and opinions in endless and frank discussions leading to the ideas advanced in this paper. In particular, R. U. Brumblay, W. C. Cooper, D. M. Coulson, N. F. Hall, H. V. Malmstadt, V. W. Meloche, and J. Pitts have given very helpful comments. This acknowledgment does not infer complete agreement by these people with all the statements made in this paper.

ALBIN KURTENACKER

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THE *Zeitschrift für analytische Chemie* has had a long and useful career. Begun in 1862 by Remigius Fresenius,[1] it has been one of the prime activities of the famous Wiesbaden laboratory and training school.[2] The editorship of this outstanding periodical has always been a position of great responsibility. The post has been held almost continuously by one of the Fresenius line of chemists, but this tradition was broken when Albin Kurtenacker was asked to take over the rewarding but also onerous duties. He has fully justified the confidence placed in him.

Born on August 12, 1886, at Unter-Themenau (then Austria, later Czechoslovakia) Kurtenacker studied chemistry at the technical universities in Brünn and Vienna. Later he went to the technical university at Delft, Holland, where he majored in microchemical analysis under P. D. C. Kley. The doctorate was conferred at Brünn in 1911, where he also habilitated as Privatdozent in 1914. From 1915 to 1918, Dr. Kurtenacker served in the Austrian army as officer in the engineer corps. After the close of World War I, he resumed his teaching career at Brünn, and was made Professor of Analytical Chemistry there in 1923. Later he was also given charge of the inorganic division. When the Nazis took over Czechoslovakia he was dismissed because he was non-Aryan. He was called to Wiesbaden in 1946 to serve as teacher and also to assist in the editorial office of the *Zeitschrift für analytische Chemie*. He became editor in 1948 in succession to Remigius Fresenius III.[3]

Professor Kurtenacker's reputation in the purely chemical field rests chiefly on his studies of sulfur compounds, particularly the polythionates. For these he worked out rapid and reliable methods of determination, based on their reactions with cyanide, sulfite, and sulfide. With the aid of these procedures he has elucidated wide areas of the chemistry of the polythionates, particularly their formation and decomposition reactions. Usable methods for producing polythionates were found, their solution equilibria were studied, etc. His analytical methods have been adopted by workers in polythionate chemistry, and have contributed markedly to the extension of the present knowledge of this class of compounds. In connection with these studies, Kurtenacker developed or improved methods for the determinatiou and separation of sulfide, sulfite, thiosulfate, sulfate, and persulfate. His analytical procedures were collected in "Analytische Chemie der Sauerstoffsäuren des Schwefels," which appeared at Stuttgart in 1938, and was reprinted at Ann Arbor in 1944.

He made kinetic and analytical studies of the action of oxidants on nitrous acid and hydroxylamine. It is notable that the halogen oxyacids, i. e., chloric, bromic, iodic, show very different behaviors toward nitrite. In the oxidation of hydroxylamine, the kind and relative amounts of the oxidation products are determined predominantly and characteristically by the hydrogen-ion concentration of the solution. The action of vanadic acid on hydroxylamine within certain pH ranges provides a clear model of intermediate reaction catalyses. A number of hitherto unknown complex copper nitrites were prepared; a new analytical procedure for hydroxylamine and hydrazine has proved to be quite usable.

Kurtenacker's studies of the composition and the existence regions of the fluorides and double fluorides of bivalent metals disclosed, among other things, that many compounds described in the literature had no actual existence. Their sponsors mistakenly regarded silicofluorides as acid metal fluorides and assigned corresponding formulas to them.

Further studies have dealt with the determination of chromium, bismuth, fluoride, etc., and with ultimate organic analysis. The turbulent political conditions led to the loss of the data regarding a good many completed researches, and could never be published.

His competence in his special fields was recognized when he was entrusted with certain sections of the chapters on sulfur, iron, cobalt, and nickel in the Abegg "Handbuch der anorganischen Chemie." As the outstanding authority, he composed the chapters on dithionic acid and the polythionic acids in the Fresenius and Jander "Handbuch der analytischen Chemie." Similar comprehensive manuscripts have unfortunately been irretrievably lost.

[1] OESPER, R. E., J. CHEM. EDUC., **14**, 313–5 (1937).
[2] FRESENIUS, R., "The centenary of the Fresenius laboratory," *Phi Lambda Upsilon Register*, **33**, 69–80 (1948).
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FRAGMENTS OF CHEMISTRY

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3. SILICON, GERMANIUM, AND TIN

In their chemistry, the elements silicon, germanium, and tin have much in common with one another as they also have with carbon. All form hydrides, AH_4, where A is a central fourth group element; all form compounds, AX_4, with the halogens; and all form mixed compounds of the type $R_{4-n}AX_n$ where R is a hydrocarbon group or hydrogen.

With alkali metals, the elements silicon, germanium, and tin quite generally form salts of the type MAR_3. Similar compounds are formed infrequently in the case of carbon. Tin also forms compounds of the type M_2SnR_2 and more complex compounds such as $M_2(SnR_2)_n$ where n is the number of R_2Sn groups in the compound. These compounds of elements of the fourth group are of importance in that they provide the means for synthesizing chains of these atoms.

Chains of Atoms. Carbon is characterized by its ability to form chains of atoms of the type C_nR_{n+2} which ordinarily are quite stable. Chains of tin atoms have been synthesized containing as many as five atoms. These chains are not very stable; they are oxidized by molecular oxygen and at higher temperatures the Sn—Sn bonds tend to dissociate.

If we could build up chains of germanium or silicon atoms, we might expect to obtain comparatively stable compounds. Unfortunately, we lack the necessary reagents for doing this. Chains of tin atoms are built up by the reaction of the metal stannides, such as $NaSnR_3$ and Na_2SnR_2, with the halides R_3SnX and R_2SnX_2. Thus, for example, we have the reaction:

$$2R_3SnX + Na_2SnR_2 = R_3Sn.SnR_2.SnR_3 + 2NaCl \quad (1)$$

With germanium, and even more so with silicon, the halides R_3GeX and R_2GeX_2 usually ammonolyze in liquid ammonia, so that in most instances reactions cannot be carried out in that solvent. However, the germanium halides do not generally ammonolyze in the amines. Furthermore, salts such as $MGeR_3$ may be prepared by action of the metals on the digermanes $(R_3Ge)_2$.

Sodium triphenylgermanide is readily prepared by the action of sodium on hexaphenyldigermane, $(Ph_3Ge)_2$, in liquid ammonia. The digermane is prepared by reduction of triphenylgermanium bromide with sodium in boiling xylene. The metal germanide is readily soluble in ether as well as benzene and liquid ammonia. At times it is advantageous to carry out reactions in an inert solvent. It is probable that hexaphenyldigermane might be prepared by reduction of the bromide by means of lithium or potassium in ethylamine or, perhaps, better, by potassium in methylamine. Such methods of reduction require further study.

Hexaethyldigermane reacts with lithium in ethylamine to form the compound $LiGeEt_3$. However, this compound ammonolyzes to form triethylgermane and the corresponding metal amide. On the other hand, the digermane reacts with potassium to form $KGeEt_3$ which does not ammonolyze in ethylamine, doubtless because potassium ethyl amide is a much stronger base than the corresponding lithium compound. Since potassium is not readily soluble in ethylamine, reaction is slow; the reaction could be carried out much more expeditiously in methylamine in which potassium is readily soluble. It is possible that hexaethyldigermane might be prepared by reduction of triethylgermanium halide in methylamine.

Diphenylgermane, $(Ph_2Ge)_x$, (in polymeric form) may be produced in poor yield by reduction of the dibromide with sodium in boiling xylene. Diphenylgermanium dibromide does not ammonolyze in ethylamine. However, in its reduction with lithium in this solvent a well-characterized crystalline compound was not obtained. It is possible that the free diphenylgermanium group is bound up with the amine, as is the triphenylsilyl group under similar conditions. Doubtless it would be advantageous to carry out the reduction with potassium in methylamine.

Sodium reacts with diphenylgermanium in liquid ammonia to form disodium diphenylgermanide, Na_2GePh_2. Evidence indicates that the compound $NaGePh_2.GePh_2Na$ is formed as an intermediate, much as in the case of corresponding tin compounds. Since the compounds $NaGePh_3$ and Na_2GePh_2 may be prepared, and since triphenylgermanium halides and diphenylgermanium dihalides are not ammonolyzed in the amines, it should be possible to build up chains of germanium atoms much as in the case of tin. The germanium compounds are much more interesting than the tin compounds because of their greater stability.

Octaphenylgermanopropane has been prepared by the reaction of diphenylgermanium dibromide with sodium triphenylgermanide in benzene. Without doubt, longer chains might be built up by means of sodium triphenyl- and sodium diphenylgermanide.

By means of suitable reactions germanium has been coupled to silicon and to tin. The silicon compounds are the more interesting because of their greater stability. It should not be too difficult to synthesize chains of germanium and silicon atoms.

Up to now, a compound corresponding to the for-

mula $(Et_2Ge)_n$ has not been prepared. Perhaps it could be prepared by reduction of dihalides with sodium at higher temperatures or by means of sodium-potassium alloy in suitable solvents. There is a chance that it might be produced by the reduction of a dihalide with potassium in methylamine.

So far as silicon compounds are concerned, the synthesis of longer chains by systematic procedure seems rather remote. However, it is always possible that the necessary reagents might be produced by some special means.

The Ge—Ge, the Si—Si, and the Ge—Si bonds are rather stable. It follows that if suitable functional groups were introduced into the substituents of these elements, compounds might be obtained that could be conveniently manipulated according to conventional methods. Up to now, little or nothing has been done so far as fourth group elements are concerned.

Reactions with Aliphatic Halides. The alkyl halides react with salts of the type MAR_3 according to the equation:

$$R'X + MAR_3 = R'AR_3 + MX \quad (2)$$

The reaction appears to be quantitative. In the case of aromatic halides, coupling does not generally take place. However, it does take place in the reaction of sodium trimethylstannide with paradichlorobenzene. Reaction takes place as follows

$$p\text{-}ClC_6H_4Cl + 2NaSnMe_3 = CH_3SnC_6H_4SnMe_3 + 2MCl \quad (3)$$

Sodium triphenylstannide reacts with chloroform according to the equation:

$$3NaSnPh_3 + HCCl_3 = HC(SnPh_3)_3 + 3NaX \quad (4)$$

In the corresponding reaction with sodium trimethylstannide the tertiary substitution product may be formed as an intermediate, but it disproportionates according to the equation:

$$2HC(SnMe_3)_3 = 2(Me_3Sn)_2 + Me_3SnCH{:}HCSnMe_3 \quad (5)$$

The formation of an ethylenic bond between the carbon atoms in this reaction appears to be well established.

When chloroform reacts with $NaGePh_3$ in liquid ammonia, triphenylgermanylmethane, Ph_3GeCH_3, and bistriphenylgermanylmethane, $(Ph_3Ge)_2CH_2$, are obtained but no tertiary substitution compound is formed. Ammonia takes part in the reaction; bistriphenylgermanylmethane is formed according to the equation:

$$3NaGePh_3 + HCCl_3 + NH_3 = 3NaCl + Ph_3GeNH_2 + (GePh_3)_2CH_2 \quad (6)$$

In the case of the mono substitution product, Ph_3GeCH_3, two halogens are replaced by hydrogen and two moles of Ph_3GeNH_2 are formed. Substitution of chlorine by germanium fails to take place when the reaction with chloroform is carried out in ether solution; hexaphenyldigermane, $(GePh_3)_2$, and sodium chloride are the chief products of reaction.

The reactions of $NaSnMe_3$, $NaSnEt_3$, $NaSnPh_3$, and $NaGePh_3$ with CCl_4 in liquid ammonia or ether are interesting. Seemingly, no coupling takes place between the tertiary metal groups and carbon. In ether, sodium chloride is formed quantitatively along with the free dimeric group, $(R_3A)_2$. In liquid ammonia, the oxide is formed from the amine Ph_3GeNH_2 by hydrolysis during separation. This indicates hydrogenation of carbon in the sense of equation (6). However, it is not known what becomes of the carbon. From the standpoint of organic chemistry these reactions are of interest.

4. GERMANIUM-SILICON COMPOUNDS

Reaction of $NaGePh_3$ with $HSiCl_3$. While coupling of three triphenylgermanyl groups with carbon does not take place in the case of chloroform, coupling takes place readily when $NaGePh_3$ reacts with silicochloroform. In ether, reaction proceeds quantitatively according to the equation:

$$3NaGePh_3 + HSiCl_3 = (GePh_3)_3SiH + 3NaCl \quad (1)$$

Tristriphenylgermanylsilane is soluble in most organic solvents from which it crystallizes readily. The hydrogen of the silane is readily replaced by halogens and the corresponding hydroxy and amino derivatives are readily prepared. More interesting, and perhaps important, is the fact that the hydrogen of the silane may be replaced by lithium in ethylamine. The silide is stable in liquid ammonia and serves as a convenient reagent for coupling this group with other organic or organo-metallic groups. With ethyl bromide, it reacts to form the compound $Ph_3GeSiC_2H_5$. In view of the difficulty with which silicon compounds of the type $MSiR_3$ are prepared, this complex germanosilyl group might well be worthy of further study. It would be of interest to know if it could be coupled with another silyl group by the reaction of the lithium salt with the halide $(Ph_3Ge)_3SiBr$.

Reaction of $NaGePh_3$ with $SiCl_4$. Sodium triphenylgermanide reacts readily and, seemingly, quantitatively with silicon tetrachloride in ether solution according to the equation:

$$8NaGePh_3 + 2SiCl_4 = 8NaCl + 2(GePh_3)_2 + (Ph_3Ge)_2Si{:}Si(GePh_3)_3 \quad (2)$$

One-half of the germanium is recovered as digermane; the remainder is coupled to silicon in some way. This compound is obtained as a solid that has not been crystallized. Its composition corresponds closely to the formula of the doubly bonded silicon compound of equation (2). If the silicon is doubly bonded one might expect to be able to cleave one of these bonds by means of lithium in ethylamine. If this could be done, interesting derivatives might be obtained by reaction with

suitable halides. Reaction (2) is worthy of further study; indeed, in view of the stability of the Ge—Si bond in compounds of this type, other compounds of germanium and silicon might well be of interest.

5. SOME OTHER REACTIONS OF SILICON

Et_3GeH and KNH_2. Trimethylstannane reacts with potassium amide in liquid ammonia according to the equation:

$$Me_3SnH + KNH_2 \rightarrow Me_3SnK + NH_3 \quad (1)$$

With triethylsilane, reaction proceeds according to the equation:

$$2Et_3SiH + KNH_2 \rightarrow (Et_3Si)_2NK + 2H_2 \quad (2)$$

This reaction illustrates the tendency of hydrogen, attached to silicon, to react with hydrogen, attached to nitrogen, with the formation of molecular hydrogen. A similar reaction appears to take place between potassium amide and triethylgermane but it has not been studied.

Et_3SiH and Ethylamine. A most interesting reaction is that between triethylsilane and ethylamine in the presence of lithium. Reaction proceeds according to the equation:

$$Et_3SiH + C_2H_5NH_2 \rightarrow Et_3SiNHC_2H_5 + H_2 \quad (3)$$

In the absence of lithium, there is no reaction; in the presence of lithium, hydrogen is evolved. The rate of reaction increases with increasing lithium concentration but the lithium takes no direct part in the reaction, being left behind in solution when reaction is completed. This is the only known instance of a reaction which is catalyzed by electrons.

It would be of interest to know if similar reactions would occur in methylamine and ethylene diamine. The action of metals other than lithium would also be of interest. The reaction rate could be determined by measuring the rate of evolution of hydrogen.

Reaction of Na_2SnMe_2 and H_2CCl_2. One might expect that reaction would take place according to the equation:

$$Na_2SnMe_2 + H_2CCl_2 \rightarrow Me_2Sn \cdot CH_2 + 2NaCl \quad (1)$$

However, although the final compound has a composition corresponding to the formula Me_2SnCH_2, the reaction is complex; the product is a polymer whose complexity increases with time.

The first stage of the reaction proceeds as follows:

$$2Na_2SnMe_2 + H_2CCl_2 \rightarrow NaSnMe_2 \cdot CH_2 \cdot SnMe_2Na + 2NaCl \quad (2)$$

The formation of the sodium salt is clearly indicated by the orange color of the solution. On adding a second molecule of methylene chloride, the following reaction occurs:

$$(NaSnMe_2)_2CH_2 + H_2CCl_2 \rightarrow 2Me_2SnCH_2 + 2NaCl \quad (3)$$

Initially the product is a fairly fluid liquid. The lowest molecular weight obtained in freezing benzene corresponded to approximately six formula weights per mol. On standing, the liquid becomes more and more viscous and the whole mass finally becomes a glassy solid.

There is no doubt that we are here dealing with a process of continuous polymerization. This compound might prove of interest to those engaged in studying polymerization mechanisms.

Reactions of Trimethyltin. Trimethyltin, which is ordinarily a dimer, has marked reducing power. It reacts with mercuric chloride to produce mercury and trimethyltin chloride. It reacts with PCl_3, $AsCl_3$, $SbCl_3$ and $SiCl_4$, trimethyltin chloride being one of the products of reaction. Compounds are formed which contain the elements P, As, Sb, or Si, but these products have not been investigated.

Trimethyltin reacts readily with stannic chloride in benzene solution with the initial formation of a white precipitate. On standing, the solution takes on a yellow color which gradually passes over to red while, at the same time, the white precipitate goes into solution. From this solution, a red precipitate is obtained. Much the same reaction takes place in ether. The red compound is also formed on addition of $SnCl_4$ vapor to Me_3Sn care being taken to keep the reaction from heating up unduly. The red compound undergoes decomposition to some extent at 100°.

The white compound as initially formed contains approximately 65 per cent of tin. On standing overnight the compound takes on an orange color and trimethyltin chloride is formed.

The red compound seems to be a rather definite substance. It analyzes 80 per cent tin and absorbs 9 per cent of oxygen. It contains some 5 per cent of chlorine along with some hydrocarbon groups. In the preparation of the red compound by reaction of $SnCl_4$ with pure Me_3Sn under controlled conditions, Me_4Sn is produced along with larger quantities of Me_3SnCl.

Thus, the products of reaction are a red compound of high tin content, trimethyltin chloride, and tetramethyltin. It would be of interest to study this reaction using other R_3Sn groups. It would also be of interest to know what reaction would occur between stannic chloride and dimethyltin.

Reactions of Some Trialkyltin Halides. Trimethyltin iodide and bromide are sensitive to light. In the presence of oxygen, trimethyltin oxide, $(Me_3Sn)_2O$, is formed in sunlight; in the presence of water, a complex compound is formed whose composition corresponds to the formula $(Me_3SnOH)_2 \cdot Me_3SnBr$. The same compound is formed by reacting trimethyltin hydroxide and trimethyltin bromide in benzene or ether solution. The compound is quite stable and is a rather good electrolyte in acetone. Further study of complex compounds of this type might be of interest.

Product from Et_3SnBr. A considerable quantity of triethyltin bromide, which had been purified by fractional distillation and which in use appeared to be a pure product, was left stoppered in a brown bottle in the dark for seven months. At the end of that time, it was observed that a considerable crop of large monoclinic crystals had settled out on the bottom of the bottle. These crystals melted sharply at 173.5–174° and appeared to be a pure compound. However, these crystals could not be identified with any known compound.

On analysis, the crystals were found to contain 45.32 per cent of tin and 30.26 per cent of bromine. This corresponds to a Sn:Br ratio of 0.992. A number of analyses for both bromine and tin gave consistent results. Evidently, the compound contained one atom of bromine per atom of tin. If the remainder of this compound consisted of ethyl groups, this would correspond to 2.20 ethyl group per atom of tin. The presence of approximately 2 per cent of inert material would account for the discrepancy. A compound of the formula EtSnOBr would have a composition in accord with the observed analytical result.

The molecular weight of the compound in benzene by the cryoscopic method was found to be 895. This high value indicates four Et_2SnBr units in the molecule, since for high molecular weights values tend to be low rather than high. The compound is remarkably stable; it may be heated to 260° without any change of its sharp melting point.

Although the compound is only slightly soluble in liquid ammonia, it may be completely reduced by three atoms of sodium with the formation of a dark red solution. This color is characteristic of a disodium tin salt of the type Na_2SnR_2. A compound of the type EtSn (ONa)Na might also yield such a color. On adding ammonium bromide a precipitate was formed and, on evaporation of the solvent, a pasty yellow product was left behind. The compound was dissolved in ether, which was evaporated in a stream of nitrogen. The compound was left behind as a yellow film on the interior wall of the container. Approximately two-thirds of the tin was recovered in this form. The compound absorbed oxygen, taking up 13.2 per cent of its own weight. This corresponds to approximately $1^1/_2$ atoms of oxygen per atom of tin.

A SIMPLE ASSEMBLY FOR CONDUCTOMETRIC TITRATIONS

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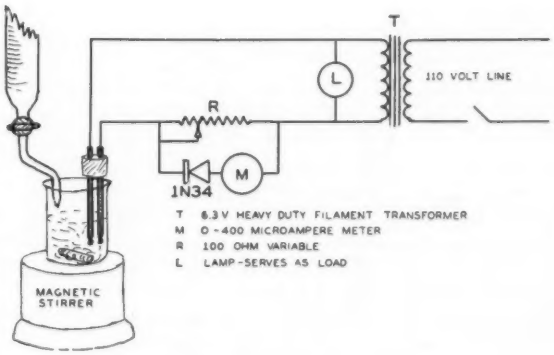
Buras and Reid,[1] have described an apparatus for conductance titrations which is sufficiently simple in operation that it may be used in the general chemistry laboratory yet capable of such precision that it is equally useful in routine work or in the physical chemistry laboratory. We have used their "circuit A" for quite some time and our only objections to it arose from our lack of large-scale a.-c. milliammeters and the frequent desire to change the meter range rather than the size of the electrodes or the distance between them. We believe that these objections are overcome by use of the circuit shown in the figure.

A husky filament transformer with a 6.3-volt 10-ampere secondary, constantly loaded by a pilot lamp drawing $^3/_4$ ampere, is used as the source of power. No noticeable difference was observed in titrations when this transformer was replaced by a Sola voltage-regulating transformer from a photoelectric colorimeter or when the entire circuit of the figure acted as part of the load on a 250-volt-ampere regulating transformer. The electrodes used are coiled platinum wires, sealed into soft-glass tubing and silver-soldered to short lengths of 12-gage copper wire. To prevent twisting



Conductometric Titration Apparatus

[1] Buras and Reid, *Ind. Eng. Chem., Anal. Ed.*, **16**, 591 (1944).

and breaking of the platinum wire in the tube the tubing was heated and filled with a black sealing wax. The electrodes are held in a fixed position with respect to each other in a two-hole rubber stopper, which is in turn held in a small condenser clamp. Magnetic stirring is used to agitate the solution; so long as the electrodes remain covered by the solution the rate of stirring seems of no consequence.

The metering arrangement, which is in series with the electrode system, consists of a variable resistance (0–100 ohms) paralleled by a germanium diode and microammeter, a Sylvania 1N34 and a 400-microampere $4^1/_2$-in. meter with an internal resistance of 800 ohms being employed. Essentially this amounts to a 0.32-volt a.-c. meter by means of which the voltage drop across the variable resistance is determined. The resistance may be a 100-ohm rheostat with a pointer and scale to allow reproduction of settings in subsequent work; we employed two decade resistances (0–10 and 0–90 ohms).

In carrying out a determination in which the resistance of the solution increases as the titration proceeds, the variable resistance is initially adjusted to a value which will cause the meter to read full-scale. In those cases in which a decrease in resistance of the solution occurs as reagent is added, we find it convenient to add the "expected excess" of titrating solution to a volume of solvent equal to the final volume of solution to be obtained, immerse the electrodes and vary the resistance to give an approximately full-scale reading on the meter. In this way the maximum amount of the meter scale is used without the danger of having the meter read off-scale before the titration has been completed. The electrodes are then removed from the solution, washed with a stream of solvent, and are ready to immerse in the solution to be titrated.

AN INEXPENSIVE THERMOSTAT

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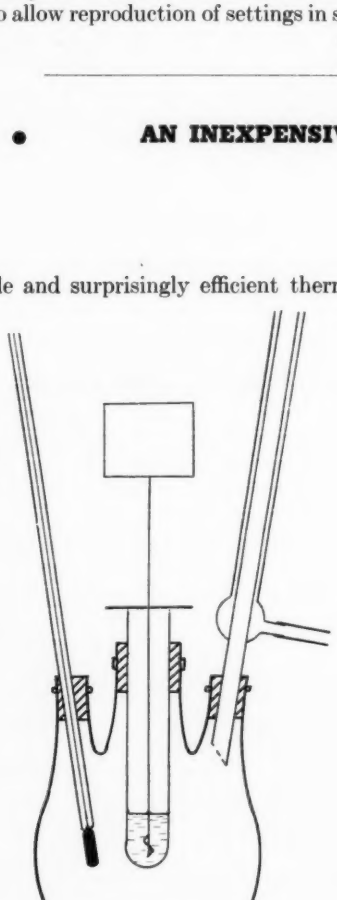
THIS simple and surprisingly efficient thermostat can be assembled in a matter of minutes. The materials are available in every laboratory, and its flexibility and accuracy rival many expensive installations. Because regulators, relays, and heaters are not needed, the setup is especially appropriate where low cost is important. In this laboratory it has been used for solubility determinations in which many different temperatures were required for short periods of time.



A one-liter, three-necked flask is fitted as shown in the diagram. The sample is placed in the test tube. Any desired temperature between about 60° and 200° can then be maintained by an appropriate selection of the boiling liquid. Mixtures can be prepared which will boil at any chosen temperature. They can be made from a wide variety of substances, and may be organic or inorganic solutions.

When a mixture of liquids is used, it has been found convenient to start with about 100 ml. of the higher-boiling liquid in the flask. The boiling is begun and the second liquid is added in small portions until the boiling point of the mixture has fallen to the desired temperature. The additions are made through the open end of the reflux condenser, and the boiling is continuous during the adjustment. The inner walls of the condenser may be flushed down by temporarily shutting off the cooling water.

During a typical run using an acetic acid-water mixture, the observed temperature was 110.0° for six hours, with a maximum variation of ± 0.1°. For greater accuracy, the thermometer can be calibrated under the experimental conditions by using a substance of known boiling point.

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CARL. W. SWEITZER
Columbian Carbon Company, New York, New York

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Each year more than 11 million pounds of carbon black reinforce and fortify paint films that protect vast values in property from the elements.

Each year nearly 17 million other pounds of carbon black serve modern civilization in a thousand ways that vary from radio communication to the clarification of fine wines, from blasting down mountains of rock to polishing fine optical parts, from opening ice-bound harbors to hastening plant growth in early spring.

So critical is the function of carbon black in rubber that you can almost say that rubber grows in carbon-black plants as truly as it does in the trees of East Indian plantations or the manufacturing plants of our synthetic rubber industry.

You could say the same of ink, of paint, of dry cells, of a great many other useful products and services.

But rubber is the most dramatic. What the carbon-black producers supply to the American rubber industry is equal to the cargo of one ship for every four that bring natural rubber around the world from Malaya, and to the output of a third unit producing synthetic rubber for every two that we have in operation. This is only the weight of carbon added to rubber of tires; its effect is magnified if we take into account the greatly extended life given tires by the carbon compounded into them. Conservatively estimated, the multiplier to use here is at least five, so that we can reasonably say 400 billion of the 500 billion miles of vehicle travel Americans enjoy annually are made on *carbon!* There is some uncertainty about that multiplier, because today no one builds automotive tires without taking advantage of longer service life provided by carbon reinforcement. That leaves us without a good modern tire made without carbon to use for comparison. As a matter of fact, synthetic rubber (GR-S, that is) is much too weak to use in tires at all until it has been strengthened by carbon, and natural rubber, too, needs the enhanced qualities given it by carbon to help it withstand the wear and tear of modern driving.

Carbon black, strangely enough, owes its most important values in tires and in most other products neither to its color (black) nor to its composition (carbon). Then, too, when we apply a simple, singular term, carbon black, to this family of tremendously useful and varied materials, we suggest that they are a single substance with a single set of properties. That is far from the fact. Thus even the name for this highly versatile and variable "soot" involves us in paradoxes and inconsistencies. We can only find our way to the truth about these matters if we examine carefully these carbons and what they do. It will help to give them a family name that is more in keeping with their properties and usefulness. Colloidal Carbons fits them better. For carbon blacks are primarily useful because they are made up of infinitesimally fine particles, far finer than those of any other commercial product and well down in the colloidal range. These extraordinarily minute particles of carbon possess by their very minuteness aggregate surface areas so huge as to be barely credible, acres and acres of surface in every pound of carbon! Finally the vast spreads of surface available in these remarkable kinds of soot, valuable though mere surface is of itself in colloids, take on special value from the peculiar nature of the carbon surface and also from the changes we can induce in it while the carbon particles are being formed.

Carbon in this fine colloidal form constitutes the soot from hydrocarbon flames where intense heat or incomplete combustion cracks up the hydrocarbon molecules and strips off their hydrogen. The ancient Chinese knew how to catch the soot from the flames of candles and oil lamps by letting the flames strike cold surfaces. They also knew how to pound up the soot with glue to make "India" ink that would give them permanent writings. Vitruvius, writing in Rome about 80 B.C., describes how the Romans controlled fires of pitch pine to make them smoke and then caught the soot in marble chambers to make long-lasting inks. Probably people used soot in this way long before that, because the oldest Egyptian hieroglyphics on papyrus that we know were written with carbon inks. But while these ancient people did their work so well that we can still read their writings, much of what they did grew out of accidental instead of thoughtful research.

Today we know a great deal about carbon and carbon particles and what makes them useful. We know how to vary the properties of the carbon product by selecting the raw material, by controlling conditions during the reaction that forms the particles, and by choosing one or another of several methods of getting the product out of the flames. Without attempting to detail all the variables involved, we can suggest these basic controlling factors: hydrocarbon raw material, proportion of oxygen available in the combustion zone, temperature at various points in the process, and time of exposure of the carbon particles to these conditions. The result

is a great number of carbons differing from each other in properties that vary by gradual increments throughout the family. Indeed, these variations are so systematic that workers in this field commonly refer to the family as the Carbon Spectrum. Table 1 shows the properties of commercially important members of the family as related to raw materials and to the processes that make them.

We need carbon for a great many purposes besides tires. Some of them we have noted and we have noted too that colloidal carbon is a paradoxical stuff. We depend largely upon it for communications, whether printed, typewritten, penned, or electrically transmitted or recorded. One of our most protective coatings to save our belongings from the elements is paint charged with colloidal carbon. Carbon is used to improve the insulating value of rubber or to make rubber a conductor that will lead harmlessly away the static charges that accumulate on airplanes, automobiles, and anesthesia bags. Carbon is essential for high-resistance radio resistors and for the conducting electrodes of arc lamps. It clarifies transformer oils and lightens fine wines, but in camouflage it darkens night-flying planes into invisibility. Colloidal carbon improves the strength of cements, but it also prevents the fine particles of powders and pigments from sticking together in solid masses. It impedes the transfer of heat through hot bottles with an effect somewhat like that of a vacuum, but it also attracts heat from the sun's rays to melt Arctic ice, or to promote the growth of plants. Colloi-

TABLE 1

A Perspective View of the Primary Properties of Typical Colloidal Carbons

The Carbons		*Approximate primary properties*[a]								
Class	*Example*	*Diameter (mμ)*	*Surface (acres/lb.)*	*Oil absorption*	*Structure index*	*Iodine adsorption*	*Roughness factor*	*% Volatile*	*pH*	*% Acetone ext.*	*% Ash*
				The Impingement Channel Process—Gas[b]							
Color	Super spectra	13	22	30	105	375	200	8	4.2	0	0.1
Color	Superba	18	16	22	110	350	250	10	3.5	0	0.01
Color	Excelsior	25	12	14.5	105	155	150	5.5	3.7	0	0.02
Color—ink	No. 140	27	11	13.5	105	120	125	8	3.2	0	0.1
Rubber—MPC	*Micronex std.	28	11	13	105	100	110	5.5	4.4	0	0.1
Rubber—EPC	*Micronex EPC	29	10	13.5	110	85	105	5	4.8	0	0.05
				The Impingement Roller Process—Gas[b]							
Color	Neo spectra II	13	22	33	110	335	180	15	3.0	0	0.02
Flow—ink	Peerless	25	12	13.5	100	170	170	12	2.8	0	0.04
				The Furnace Process—Gas or Gas Plus Oil[c]							
Rubber—VFF	Statex K	33	9	13.5	130	85	110	0.5	9	0.05	0.8
Rubber—FF	*Statex B	43	7	9.5	100	50	85	0.5	9.3	0.1	0.8
Rubber—HMF	Statex 93	58	5	10.5	120	30	75	0.5	9.5	0.2	0.5
Rubber—SRF	*Furnex	85	3.5	8.5	105	25	85	0.5	10.0	0.1	0.5
				The Furnace Process—Oil or Oil Plus Gas[c]							
Rubber—HAF	Statex R	28	11	15	150	80	85	1.0	8	0.1	0.2
Rubber—FEF	Statex M	58	5	15.5	180	40	95	0.3	9.5	0.1	0.5
				The Thermal Process—Gas[d]							
Rubber—MT	P-33	150	2	5	65	40(25)	235(150)	0.3	8.5	1.5	0.1
				The Acetylene Process—Gas[d]							
High structure	Shawinigan	45	7	38	400	65	110	0.3	6.5	0.1	0.01
				The Lampblack (Germantown) Process—Oil[e]							
Color	LB DD	65	4.5	16	190	20	55	10	3	3.0	0.1
Rubber—RMV	LB #18	100	2.5	15	200	15	70	1.5	3.5	0.2	0.12

* Ink carbons falling within these ranges of properties include, respectively, Raven 11, Raven 15, Statex B-12, and Molacco.

[a] *Property Values* are given as modal for the class. The diameter and specific surface values are based on electron microscope measurements. Oil absorption, expressed in gallons per 100 lb., in conjunction with particle size, indicates the extent of reticulate chain structure present, the index for which is given by Structure Index (normal as 100) (*10*). Iodine adsorption provides a measure of total surface including porosity, the index for which is given by Roughness Factor (normal as 100) (*12*). Volatile and pH are determined by standard industry methods. Per cent acetone extractable indicates the oiliness remaining on the particle surface. Per cent ash is usual test.

[b] *The Impingement Process* employs many small flames which impinge on channels or rollers that collect the carbon particles from the flames. These small flames produce the finer carbons with the range of this fineness controlled primarily by flame size and temperature. Since the air to gas ratio is very high these carbons become surface oxidized, leading to high volatile and acid pH properties. The longer exposure to air in the roller process produces more oxidation. As no water is used for cooling the ash content is very low. Since the flame is small and steady the structure remains constant.

[c] *The Furnace Processes* employ, in contrast, relatively huge flames confined in refractory chambers or furnaces, from which flames the carbon particles are released as an aerosol. In all cases these colloidal particles are collected by the successive steps of water-cooling the gases, flocculating the particles electrostatically, and causing the floccules to settle out in cyclone chambers. Since the ratio of air to hydrocarbon is low, the atmosphere is neutral to reducing and little surface oxidation develops (low volatile). The water cooling introduces water salts and so raises ash. Traces of oily impurities normally remain on the particle surface. Fineness and structure are controlled by varying the flame size, the introduction and composition of the hydrocarbon feed, the reaction velocity, and the reaction temperature.

[d] *The Thermal Processes* employ heated refractory furnaces in which the hydrocarbon gases are decomposed into carbon and hydrogen. In the thermal process using natural gas the reaction is endothermic, so heat must be supplied at regular intervals. This type of reaction leads to coarse, low structure, oily carbons. In the acetylene process using acetylene gas the reaction is exothermic and yields somewhat finer, higher structure, nonoily carbons. Since air is absent in both processes the oxygen volatile is negligible. Ash content is low since little or no water is used for cooling.

[e] *The Lampblack Processes*, burning oil in open pans, by a free-flame method, give relatively coarse carbons of high structure. The high air ratio leads to oxygen volatile and acid pH. Traces of oily impurities normally remain on the particle surface. Ash is normally low since no cooling water is used.

dal carbon powder feels soft, yet it is the abrasive used after the finest rouge for polishing the lenses, prisms, and reflectors of precision optical instruments, and it is employed in making the hardest tool-steel alloys. Colloidal carbons are usually considered chemically inert, largely because they are insoluble in water or any other customary solvent, and hence are incapable of entering the kinds of chemical reactions that are usual at room temperature. But the standard quality carbon black made by the channel process for the rubber industry (EPC) can impart to water or salt solution in which it is suspended a pH of 4.5, about the acidity of lemon juice! Since each pound of this type of carbon has about ten acres of particle surface, wherein resides this acid quality, we are justified in saying that its acidity is equivalent to an extremely thin layer of lemon juice spread evenly over ten acres, and that is far from "chemically inert." Furthermore, colloidal carbon dipped in liquid oxygen forms one of our most powerful yet safest explosives.

This diverse utility of the colloidal carbons is based on different combinations of useful, fundamental properties that employ every characteristic of the ultimate discrete particles of carbon. The particles that interest us here are the discrete solid particles that have independent physical existence. They are visible and measurable in electron micrographs. They are many times larger (even 100 times larger) than the minute crystallites that constitute the particles here intended. The crystallites are formed by the orderly arrangement of carbon atoms in crystal lattices, and they can be detected and measured only by X-ray methods. We are not concerned here with these crystallites but only with the particles formed by their aggregation.

The colloidal size of colloidal carbon particles enables them to reinforce rubber. The shape or structure of the reticulate chains they easily form produces the stiffening effect noticed in many applications. Their color (blackness) has wide usefulness in a multitude of various products ranging from automotive finishes and printing inks to caviar. The surface activity of the particles of colloidal carbons gives them value as purifying and healing agents. And their composition (carbon) is the basis of a variety of applications from tool tips to foam expanded glass.

The behavior and value of any colloidal carbon is determined by the characteristics, both physical and chemical, of its particles. We have noted some of the effects produced by these characteristics of carbon particles and something of the way that they can be controlled during the process that makes the carbon particles. It is possible to apply after-treatments to the carbon that will cause other variations in properties, but practically all the different colloidal carbons used in industry are made to specification in the first place without after treatment. We can name half a dozen properties of carbon particles that together, along with color and composition, establish the use patterns of the various colloidal carbons. The physical properties consist of: surface area, structure of particle aggregates, and porosity. The chemical properties are: the presence of oxygen-volatile matter (as distinct from the oily volatile matter of residual hydrocarbon), unsaturation or free chemical bonding forces at the surfaces, and the presence of alkaline salts introduced during manufacture. Each of these is so important in understanding the colloidal carbon picture that they justify separate individual discussion.

Surface Area. We can see from electron micrographs that have been made of various types of colloidal carbons that the ultimate particles are spheroidal in shape. We can also measure the dimensions of these particles in the micrographs and from the known magnification calculate the actual dimensions of the particles themselves. Having these dimensions, we can assume the particles to be spheres and calculate the volumes and surface areas within a very close approximation of the true values. Actually, the error we introduce by our assumption is negligible and well within the limits of error of the original measurements. Once having arrived at the surface and volume of each particle, we can very simply take the next step and, using the known density of carbon itself, estimate the number of particles per pound of the particular carbon and the aggregate surface area of all of these. The magnitude of these two figures is staggering. (See Table 1.)

Even before the electron microscope allowed us to photograph and measure accurately the individual particles of carbon black, their colloidal nature had been well established. For example, the particle diameter of Micronex carbon black was reported to be: below 80 mμ by the microscopy method of Bernard (*1*); between 50 and 60 mμ by the ultramicroscopic count methods of Bernard (*1*), Peterfi (*2*), and Gehman and Morris (*3*); and between 45 and 68 mμ by calculations from pore radii (*3*). Electron micrographs (*4*) later showed that the diameters of the discrete spheroidal particles of Micronex carbon black range from 10 to 50 mμ, with the mean and modal values lying between 25 and 30 mμ. Using their low temperature adsorption method, Emmett and DeWitt (*5*) later calculated the average diameter from adsorption data to be 31 mμ. The electron microscope method revealed mean values for the other members of the carbon-black family (*6*) ranging from 13 mμ for the finest color carbons to 100 mμ for coarse lampblacks and even higher values for thermal carbons (see Table 1).

This extremely fine subdivision of colloidal carbons is easily translated, as we have noted, into units of acres of particle surface per pound of carbon and this unit relates fineness to area that is the vital factor in many of its uses. When incorporated into rubber compounds, for example, the enormous surface of the carbon particles (10 or so acres per pound of carbon) reinforces and toughens the rubber, giving it greatly improved resistance to abrasion and tear. The magnitude of this reinforcing effect for all the important rubbers is shown in Figure 1 in terms of increased tensile strength (*7*). The high mileage of modern automobile tires is chiefly due to this reinforcement of rubber by colloidal particles of carbon.

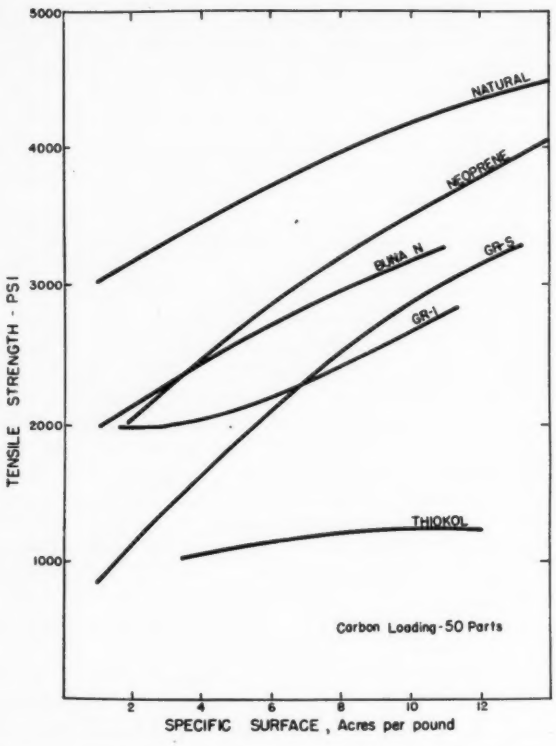


Figure 1. Effect of Colloidal Carbons on Tensile Strength of Various Rubbers

To a corresponding extent the strength and usefulness of a multitude of other rubber products has been improved through the use of colloidal carbons.

This reinforcing effect of colloidal carbons is also applied in other compositions; a few of these are listed in Table 2.

Structure of Particle Aggregates. Some members of the carbon-black family behave as if their particles were of a different size from that measured. Particularly, they possess greater absorption of liquids and enhanced stiffening effects in rubber compounds. This fact was recognized in 1941 (*6*) as possibly indicating that these particles possess a pronounced anisotropy, "a clustered pigment structure or disposition consisting of carbon to carbon complexes or networks." In 1945 (*8*) the electron microscope solved this mystery. Three-dimensional electron micrographs revealed that to varying degrees the particles of all carbons as formed exhibit reticulate chain structures. The persistence of a goodly proportion of this structure through processing explains much of the anomalous behavior of these carbons. This property is by common consent designated "Structure," although a more recent investigation refers to this property of carbons as the "Shape Factor" (*9*).

One effect of this joining of particles together in reticulate chains is to reduce the total exposed surface by a small amount, 20 per cent at most. Hence it is unimportant in surface and in chemical adsorption. But in absorption of liquids, these structure units exert a profound effect. They provide a spongy mass of carbon that will hold, sponge-fashion, large volumes of liquid. The absorption range for carbons having 50-mμ particles, for example, extends from less than 10 gallons per 100 lb. of carbon to more than 30 gallons, an increase of more than 200 per cent. This high liquid capacity of the high-structure carbons is the basis of many important applications (Table 3).

A convenient quantitative measure of this structure effect is expressed as the "Structure Index" (*10*) by values based on absorption of linseed oil. The range of the structure index of the various types of colloidal carbons is shown in Figure 2. Commonly higher structure carbons are made by processes employing oil, and lower structure carbons by

TABLE 2
Uses of Colloidal Carbon Based on Fineness as Primary Property

Product or process	*Function*	*Mechanism*
Rubber products	To reinforce or toughen rubber for max. service (for various rubber products carbons are selected to provide balance between reinforcement and other properties)	Not fully understood but probably involves a carbon-to-rubber bond via one or a combination of the following mechanisms: (1) Van der Waals forces (2) Chemical bonding (3) Sulfur bridging (*21*) (4) Carbon gel complex (*25*)
Passenger tire treads	Max. wear, min. cracking, good processing	
Truck tire treads	Max. wear, min. cracking, min. heat build-up	
Tire carcasses and inner tubes	Reinforcement, hot tear resistance, min. heat build-up	
Tank tracks	Max. wear, resistance to chipping	
Footwear	Max. wear, min. cracking, good snap	
Conveyor belting	Max. resistance to wear, tear, cutting and cracking	
Hose	Max. wear, min. cracking, good extrusion	
Wire jackets	Max. wear, ozone resistance, good extrusion	
Vibration mountings	Reinforcement, low compression set, min. heat build-up	
Gaskets and seals	Reinforcement, low compression set	
Brake linings	Max. wear, min. grabbing, even friction stresses	
Rubber to metal cements	Reinforcement, control of cure	
Nonrubber products		
Grinding of portland cement	Aids grinding of clinker	Prevents caking of cement particles
Sulfur cement	To strengthen and to improve workability	Possibly physical bonding
Dusting sulfur	To prevent caking of sulfur and to make it free-flowing	Acts as a separating medium

processes employing gas (Table 1). The extremes are made by the thermal process, the lowest structure by the endothermic process from methane (P-33) and the highest structure by the exothermic process from acetylene (Shawinigan). It is apparent that the nature of the hydrocarbon used, as well as the process, is important in developing structure.

This structure property for colloidal carbons has many important uses primarily for its effect in stiffening or controlling the viscosity of compounds (Table 4). Examples include the anomalously high stiffening effect in rubber, matte or dull finishes in inks and in paints, and absorption of relatively large volumes of liquids in dry cells (electrolyte solution) and explosives (liquid oxygen).

Porosity of Particles. Measurements of the adsorption of nitrogen at low temperatures (*11*) by rubber blacks made by the channel process and for some of the furnace blacks gave values for their surfaces in good agreement with the electron microscope observations. Note that we are here concerned with surface adsorption and not mass absorption. But the nitrogen method gave values for carbons of high volatile content and of high color that are consistently high. This suggested that the surfaces of these high-volatile carbons may be porous or pitted, an effect designated later as "Roughness Factor" (*12*). It is also known as particle porosity and while it substantially increases the surface area per pound of certain carbons, the effect of this added surface is more pronounced with media of low molecular weight (nitrogen, water, iodine, etc.) than it is with materials having large molecules of complex structure (rubber and plastics). Presumably the reason for this lies in the dimensions of the pores and of the adsorbed molecules. The surface increment from this porosity or roughness is far too great to be neglected under most practical conditions and may amount to as much as 600 per cent added to the surface, calculated from electron micrographs. Its most important effects appear in phenomena involving the adsorptive characteristics of the carbon particles.

TABLE 3
Uses of Colloidal Carbon Based on Adsorption as Primary Property

Product or process	*Function*	*Mechanism*
Telephone cables	Carbon black in paper insulation adsorbs electrolytes and moisture from cable oil	Complete trace adsorption
Transformer oils	Improve dielectric strength by adsorbing electrolytes and moisture	Complete trace adsorption
Wire insulation (rubber)	Same type as above	Complete trace adsorption
Radio tubes	To remove gas traces	Complete trace adsorption
Rare metals recovery	To recover precious metals from waste photographic solutions, etc.	Selective cationic adsorption
Wine	Decolorizes	Complete trace adsorption
Oils	Decolorizes or deodorizes	Complete trace adsorption
Physiological	To remove toxic bodies	Complete trace adsorption

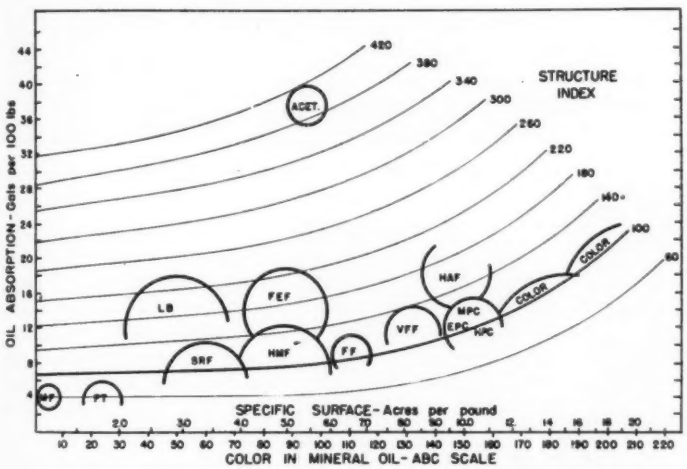


Figure 2. Structure Index for the Family of Colloidal Carbons

Oxygen-Volatile Matter. The significant effect of oxygen-volatile matter (as contrasted to that of volatile hydrocarbons) in carbon on the adsorptive behavior of colloidal carbons in rubber compounds was investigated in the 1920's. Goodwin and Park (*13*), Beaver and Keller (*14*), Johnson (*15*), Wiegand (*16*), and others studied the problem. They generally agreed that part of this combined oxygen is present on the carbon surface in form of carboxyl groups (74

TABLE 4
Uses of Colloidal Carbon Based on Absorption as Primary Property

Process or product	*Function*	*Mechanism*
Sealing compounds	To control viscosity and strength	Stiffening or gelation of system
Roofing asphalt	To prevent flow	Stiffening or gelation of system
Silicone oil lubricants	To control viscosity	Stiffening or gelation of system
Greases	To control viscosity and prevent flow	Stiffening or gelation of system
Dry cell batteries[a]	To prepare stiff depolarizer paste	Absorption of electrolyte solution

[a] In this application electrical conductivity is rated as primary property.

per cent inert, 26 per cent acidic, according to Villars (*17*)). Being acid in their reaction and pH effect (*18*), these carboxyl groups selectively adsorb or remove alkaline adsorbates from solutions and compounds. Among these are the alkaline accelerators or activators of vulcanization in rubber (*19*). Removal of these by adsorption retards cure of the rubber, and similarly, adsorption of metallic driers in paints retards their drying (*20*). We can readily correlate volatile matter and pH of the carbons with alkaline adsorption if we allow for the surface adsorption that is also involved. The magnitude of this effect varies with the particle size of the carbon and with its volatile content. Peerless carbon with about 15 per cent volatile matter shows an alkaline adsorption divided equally between surface effect and carboxyl groups. With the finer carbons having the same volatile content, surface has a proportionately greater effect.

Numerous theories have been advanced to explain this adsorption of alkaline salts by the oxygen-volatile carbons, but the most plausible involves simple ion exchange, or neutralization, with the cation replacing a hydrogen ion either in a carboxyl group or on the carbon surface. This theory also explains the well-known effect of the acid carbons in removing metal ions from solutions of neutral salts with a resultant increase in acidity, and the converse effect of the alkaline carbons in removing anions to increase the alkalinity of the solution. These effects are sometimes referred to as "hydrolytic" adsorption.

Chemical Unsaturation of Surfaces. Adsorption curves of colloidal carbons follow the Freundlich adsorption equations, as shown in Figure 3. In this group of curves, we can separate the effects of particle surface, particle porosity, and oxygen-volatile. The difference between the curves on the right and left of the graph is due to oxygen-volatile. Deoxidation of Peerless and Neo Spectra II (see Table 1) containing high oxygen volatile causes a drop of about 50 per cent in adsorption. Furnace carbons containing less than 1 per cent oxygen-volatile are practically unaffected by the same deoxidation treatment. Deoxidation does not reduce the adsorption of either the very fine carbons (Neo Spectra II, Super Spectra) or the high-volatile carbons (Peerless) to their proper positions in the particle-surface scale. Compared with Statex and Micronex, these very fine carbons (*i. e.*, Neo Spectra II and Super Spectra) are porous and have pronounced adsorption due to this particle porosity. This porosity characteristic of the high-volatile carbons has been

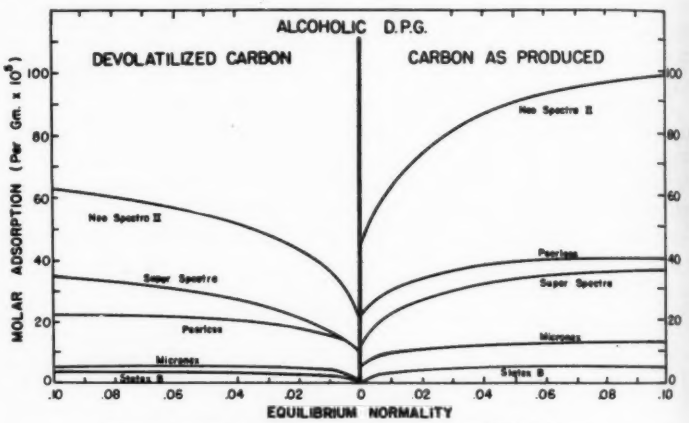
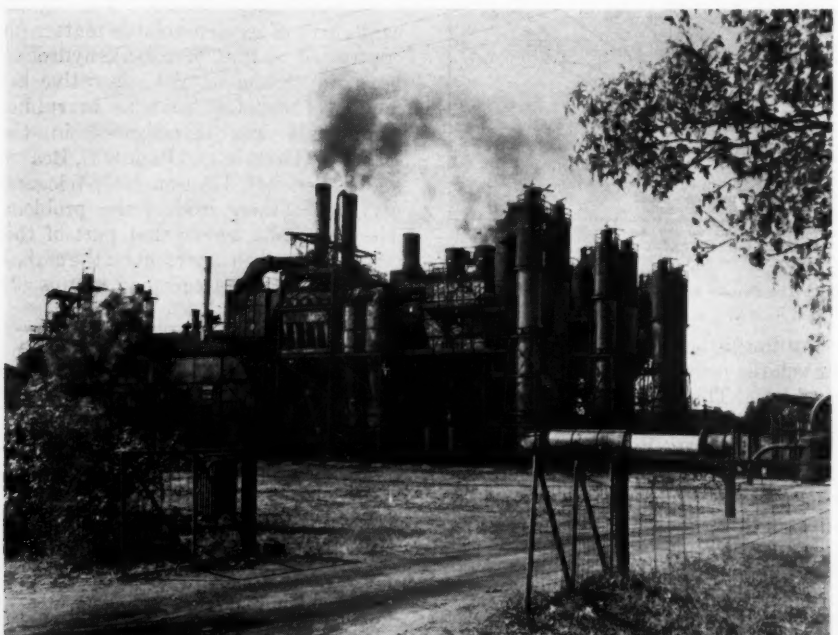
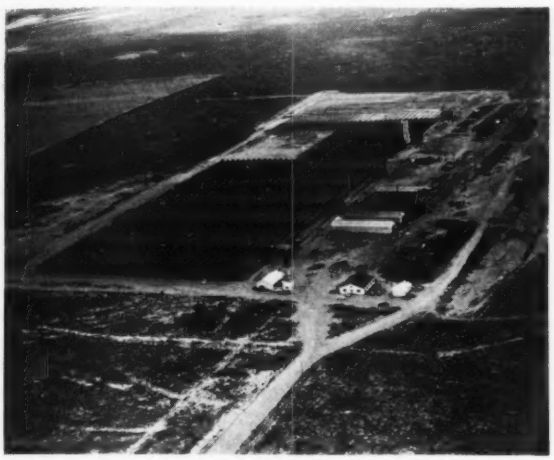


Figure 3. Typical Adsorption Isotherms of Colloidal Carbons



The furnace process for producing colloidal carbon from gaseous or liquid hydrocarbons, or mixtures of the two, is carried out in this plant.

pointed out by Smith (*11*) and others (*12*). Another significant feature of these curves is the location of the zero values well up on the ordinate scale. This indicates complete removal of the adsorbate at low concentrations, possibly by chemisorption, a phenomenon which has been put to practical use in many applications. These curves are typical and have similar shapes (but differing values) with any adsorbate or any carbon. In the presence of several adsorbates,



The channel process for making colloidal carbon is operated in rows of galvanized iron burner houses like these.

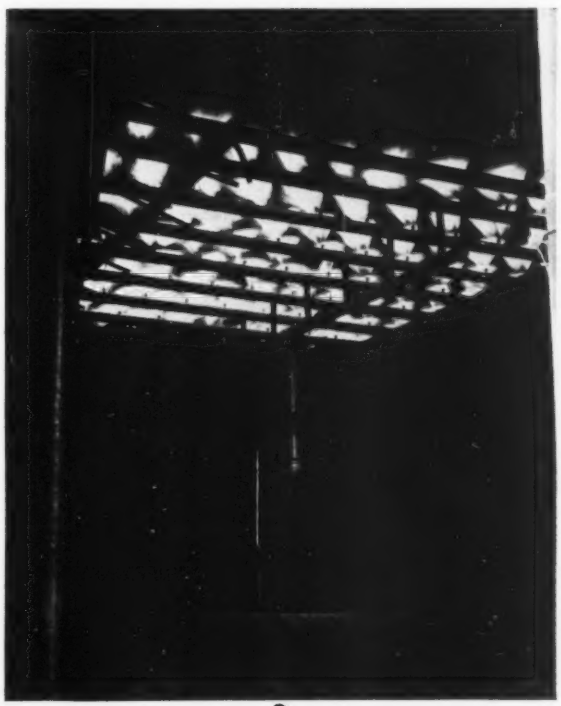
Traube's rule is found to hold for inorganic as well as organic solutes, an important phenomenon in applications involving recovery of heavy metal ions from complex aqueous wastes.

The oily thermal carbons, and to a lesser degree some of the oily furnace carbons, show a much higher iodine adsorption than is required by their total available surface. The assumption is that during manufacture in a highly reducing atmosphere these residual oils develop a chemical unsaturation which takes up iodine. This effect may be particularly strong if the adsorption is measured in a solvent like benzene which extracts the oils from the carbons. If these oils are removed before testing the carbons, the iodine values still remain too high, indicating a similar unsaturation on the particle surface. If these extracted blacks are oxidized to satisfy this unsaturation, the iodine adsorption values then fall in line with those calculated from particle surface. This unsaturation probably consists of ethylenic double bonds that, according to Stearns (*21*), are present on all carbon surfaces. Further studies are required to determine whether halogens are adsorbed by colloidal carbons only at double bond sites or over the whole surface.

Alkaline Salts. The presence on furnace blacks of alkaline salts from the water used for spray cooling during manufacture is also a factor in adsorption. These salts, largely carbonates, may be present on the carbon in quantities ranging from 0.1 to 1.0 per cent and naturally affect the behavior of these carbons toward acid solutes in aqueous media.

Adsorption Applications. The adsorption behavior of colloidal carbon probably has more ramifications than any of the other properties of this amazing colloid and is less understood. However, recognition of the basic factors described, together with some understanding of the mechanism of adsorption, aids greatly in rationalizing the importance of adsorption in the manifold applications of colloidal carbon.

Adsorption is a major factor in rubber reinforcement and important but secondary in most of the nonrubber applications of colloidal carbons. A number of other interesting uses are based upon the primary and direct role of adsorption. The complete removal of traces of electrolytes by colloidal carbons (Figure 3) is the basis for their use in "cleaning up" trace impurities in dielectrics. The magnitude of the total adsorptive effect, equivalent for some colloidal carbons to that of adsorbent chars, is applied in clarifying, deodorizing, and purifying processes. Selective adsorption of metallic cations is utilized in the recovery of rare or valuable metals from industrial wastes. This adsorptive removal of trace impurities, particularly those of high molecular weight, has led to the study and use of colloidal carbons to remove and deactivate toxic and physiologically harmful bodies from body fluids. Some of these adsorption uses are noted in Table 3.



These natural gas flames impinge upon iron channels that move slowly back and forth through them to collect the colloidal carbon as formed.

Blackness or Radiation Absorption. Blackness, or color, is the historical, best known, and most widely used property of colloidal carbons. Colloidal carbons possess high absorptive power for light in the range of the visible spectrum, as high as 98 per cent, and thus they are the most satisfactory black pigments for most purposes. The chemical stability of these carbon pigments gives them extreme permanence; the hieroglyphics of ancient Egyptian manuscripts written with carbon have withstood the ravages of thousands of years.

Fineness of subdivision is the dominating factor in the absorption of visible light and hence in blackness of colloidal carbons. It ranges from a dark bluish gray (caused by high reflectance of coarser particles), to an intense black with a brownish cast (from the scattering of the blue wave lengths by the smaller particles). This brown color is especially evident in the light transmitted by dilute colloidal dispersions of the finer carbons. Photometric methods are fairly satisfactory for the quantitive evaluation of blackness (*22*), but the most sensitive method is direct visual comparison under the arc light (*23*). Visual comparison is made with a series of color standards assigned values of shade. The coarsest and grayest commercial carbon is assigned a value of zero (*10*). The complete color range comprises 260 shades, equivalent to a fineness range of 1 to 100 millimicrons diameter corresponding to 1 to 31 acres of surface per pound of carbon as derived from electron microscope measurements.

Almost as important for blackness as fine particles is optimum dispersion of carbon in the vehicle; the greater the dispersion, the more intense the blackness. The increase in intensity obtained through complete dispersion of the finer carbons may be great enough to give an entirely new order of blackness (*23*).

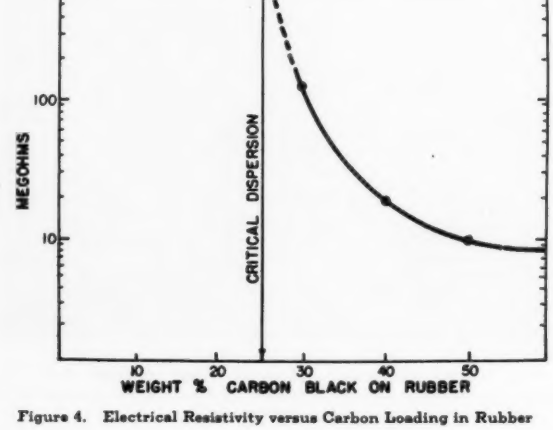


Figure 4. Electrical Resistivity versus Carbon Loading in Rubber

The tremendous surface developed through dispersion of colloidal carbons gives them covering or hiding power that surpasses all other pigments in protective films and in printed surfaces. Hiding power and, to a lesser extent, solid color or mass tone are also influenced by the nature of the coated surface, the irregularities of the film, the distribution of the pigment in the film and the refractive index of the coating film (*24*).

The high absorption by colloidal carbons of visible light also extends to the shorter wave lengths of the ultraviolet and to the longer (heat) wave lengths of the infrared. Since ultraviolet promotes film deterioration, colloidal carbon pigments protect the film against aging. This screening of the ultraviolet accounts for the extreme durability of black protective coatings outdoors as in automotive finishes, industrial paints, etc. (See Tables 5 and 6).

Electrical Conductance. Since carbon is a good conductor of electricity it is not surprising that many uses of colloidal carbon should be based on this property. Its ultrafine particles allow this conducting property to be varied within wide limits, through choice of carbon type and loading. The physical and chemical properties of the carbon particles profoundly affect electrical conductance.

Oxygen-volatile or oily impurities on the surface of the carbon particles may increase resistivity a hundredfold. Thus heat treatment of the product is often needed to "clean" the surface to minimize contact resistance and hence raise conductance. The conductivity of "clean" particles follows particle size; the finer the particles, the greater the conductivity (*10*). In a nonconducting vehicle, such as rubber, maximum conductivity is obtained short of ultimate dispersion of discrete particles, since ultimate dispersion insulates the particles from each other by the vehicle. The desirable state is uniform distribution of aggregate units to provide maximum carbon bridges throughout the matrix.

Carbon loading (*i. e.*, the proportion of carbon to vehicle in pounds per 100 pounds) is important in all dispersions of colloidal carbons in nonconducting vehicles, such as rubber. Low loadings have the insulating properties of the vehicle and the conductivity of the carbon becomes effective only when loadings reach approximately 25 per cent (for a Micronex carbon) (*25*). This is shown in Figure 4. This critical loading varies with particle size, being lower for the finer carbons and higher for the coarser ones. The increase in resistivity shown in this graph for the lower loadings is ascribed to the adsorptive removal of conducting electrolytic impurities by the carbon.

Carbons must be selected or modified to give the conductive property desired. In rubber goods, for example, reinforcement is combined with conductance, so that reinforcing carbons are selected which have surfaces sufficiently "clean" to give the desired conduct-

ance. In radio resistors the carbon must be selected or modified to give a zero temperature coefficient of resistivity. In dry cells the carbon must not only develop maximum conductivity but must also hold the electrolyte solution in the depolarizer paste (the bobbin). A few of these more important applications are shown in Table 7.

Chemical Reactivity. Carbon is a well-known chemical reducing agent in the metallurgical industries. In many metallurgical operations, especially those involving the harder and rarer metals, a carbon with minimum particle size gives the most efficient reduction and for this purpose colloidal carbon is ideal. The powdered ore is intimately mixed with colloidal carbon and the reduction carried out to yield metal of great purity. Or the reduction with colloidal carbon may be a refining operation to improve the purity of the metal. In either case carbons of exceptional purity are advantageous.

Colloidal carbons are also extremely important in certain reactions forming carbon compounds, such as the carbide reaction in a hydrogen sintering furnace to

TABLE 5
Uses of Colloidal Carbon Based on Blackness as Primary Property

Product or process	*Function*	*Mechanism*
Inks		
Newspapers, books, magazines, etc.	To provide permanent black record of printed or written word, with maximum covering power	Absorption of visible spectrum
Typewriter ribbons		
Carbon paper		
Mimeograph		
Writing and drawing (India)		
Protective and Decorative Coatings		
Paints, enamels, and lacquers	To provide permanent protective black coating or finish	Absorption of visible spectrum for blackness and ultraviolet spectrum to protect film
Organisols, plastisols		
Latices (rubbers, resins), emulsions		
Wallpaper		
Leather finishes	To provide black color	Absorption of visible spectrum
Shoe and stove polishes		
Tinting colors	To produce grays and to shade other colors	
Paper and Board Compositions		
Black and gray paper	To provide permanent black color	Absorption of visible spectrum
Cardboard		
Photographic paper		
Panel board		
Blackboard		
Products for Industry, Home, and Farm		
Plastics (molded or extruded)	To provide black coloration of permanence (textiles, etc.), of purity (foods), of colloidal fineness (synthetic fibers), etc.	Absorption of visible spectrum
Linoleum		
Phonograph records		
Cement and mortar compositions, concrete roads		
Textiles (printing)		
Synthetic fibers (integral coloring)		
Candy and food		
Cosmetics		
Crayons		
Glass (brown and black)		
Fertilizers		
Insecticides		

TABLE 6
Uses of Colloidal Carbon Based on Radiation Absorption as Primary Property

Process or product	*Function*	*Mechanism*
Polyethylene and polystyrene wire and cable jackets	To prevent breakdown of resin by sunlight exposure	Absorption of ultraviolet radiation (ultraviolet screening)
Photographic film backing	Antihalation	Absorption of visible and ultraviolet radiation
Paints for darkroom and optical instruments	To trap stray light	Absorption of visible and ultraviolet radiation
Thermal insulation	To act as an insulating material in place of asbestos, etc.	Absorption of infrared radiation
Solar heating	For heating homes by storing heat of sun	Absorption of infrared radiation
Harbor ice	To melt ice	Absorption of infrared radiation
Soil treatment	To speed germination of seeds by increasing temperature of soil	Absorption of infrared radiation

TABLE 7
Uses of Colloidal Carbon Based on Electrical Conductivity as Primary Property

Process or product	*Function*	*Mechanism*
Rubber tires—antistatic	To provide electrical conductivity to release static	The high structure carbons provide reticulate chains of carbon particles, forming almost continuous paths through the medium. The normal structure carbons provide carbon paths by higher loading, by selection of grade, etc.
Rubber shoes		
Rubber anaesthesia bags		
Rubber antistatic flooring		
Antistatic paints and inks		
Radiant heating panels	To provide electrical conductivity	
Plywood	To provide for dielectric heating in manufacture of plywood	
Conducting photographic paper	To release static	
Conducting tele-communication paper	Electrical conductivity	
Radio resistors		
Radio tubes	To trap stray electrons	
Motor and generator brushes	Electrical conductivity	
Contact carbons		
Anodes		
Dry batteries	Conductance of discharge current	
Wet storage batteries	Expander for litharge	

TABLE 8
Uses of Colloidal Carbon Based on Chemical Reactivity as Primary Property

Process or product	*Function*	*Mechanism*
Carbon steel	Added to steel to harden it	Alloying of carbon and steel
Surface hardening or carburizing of steel	Added to surface of finished steel to case harden it on heating	Iron carbides are formed
Carbides	For reaction with tungsten to form tungsten carbide	Direct combination
Metal reduction processes	For reducing ores of Ba, Be, V, W	Reduction process
Copper refining	To prevent oxidation	
Liquid oxygen explosive	To hold sufficient oxygen to form CO_2 completely on detonation	Explosive combustion
Foam glass	For the cellulation of glass in manufacturing glass insulation	Gas formation

form the carbide of tungsten or of other metals. Another case in point is the mixture of liquid oxygen and colloidal carbon to form an explosive with fragmentation power superior to TNT and that yields an end product of harmless carbon dioxide. Still another chemical reaction of colloidal carbon finds use as a gas former in the manufacture of foam glass. These and other applications are shown in Table 8. In all of these applications colloidal carbon is a unique reagent since its ultrafine particles permit an efficiency in the reaction that cannot be approached by the coarser cokes and chars. Cost alone prevents its wider use in reduction processes.

Obviously we are here dealing with an extremely valuable and versatile material of great industrial utility. Its value increases as the peculiar properties of the particles of colloidal carbon are more fully understood.

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TRIGONOMETRIC FORM OF THE LORENTZ TRANSFORMATION

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CALCULATIONS involving the various Lorentz transformations may be materially shortened by employing a trigonometric device for the evaluation of the term $(1 - v^2/c^2)^{1/2}$, which may be rearranged to $(c^2 - v^2)^{1/2}/c$.

Consider a right triangle having base v (velocity of translation) and hypotenuse c (velocity of light), calling the angle included between v and c the angle A. The third side is $(c^2 - v^2)^{1/2}$.

From the triangle it is evident that $\sin A = (c^2 - v^2)^{1/2}/c$ and that $\cos A = v/c$, so that

$$\sin \cos^{-1} \frac{v}{c} = (1 - v^2/c^2)^{1/2}.$$

Suppose, for example, it is desired to calculate the mass of a moving particle from the Einstein equation

$$m = m_0/(1 - v^2/c^2)^{1/2}.$$

Making the trigonometric substitution, it is only necessary to find the angle whose cosine is v/c and divide the rest mass by the sine of the latter angle to determine the relativistic mass.

PRIMORDIAL GLASSES

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ARCHAIC glass and glazed articles have been discovered by archeologists, and the manufacture of glass, along with lime, is probably among the earliest achievements of the handicraftsman when he appeared on earth carrying a lighted torch. The burnt brick may have been a precursor, since clay figurines, hardened by fire., have been found in the cave dwellings of paleolithic man.

DISCOVERY OF GLASS

Baked building bricks are first encountered in neolithic times. Man, subsequent to his rural pursuits, treated minerals with fire for the production of goods. With brick available he built furnaces, evolution of pure metallurgy followed. The bulk of the evidence supported by correct interpretation of ancient texts seems to indicate that glass was first produced by accidental fusion inside a furnace. Such fusion is a common occurrence in our present-day kilns—a glaze covering the inner lining, the surfaces of bricks, etc., and even glassy drops forming when excess heat unduly raises the temperature above the fusion point of lining or charge.[1] However, under similar conditions, natural stones can also undergo a glazing process; the author found among excavation debris in the Agora of Ancient Athens a number of partly glazed schistic stones. Schists, commonly known in Attica as *"granitsas"* (ganister corrupted) are still used locally in furnace construction. The stone surfacing of the so-called Vitrified Forts, built centuries ago in Scotland, Ireland, and elsewhere, was undoubtedly glazed by the only possible process, namely persistent firing, and so were the surfaces of the base of the very ancient Fort of Acragas (Agrigente, Sicily), a rock-wall patched with masonry. According to reliable information[2] the stones of the latter were scorched.

The occurrence of natural fusible stones probably advanced a Latin myth[3] concerning the accidental discovery of glass by Phoenician seafarers, who on a sandy shore started fire under a cooking pot set on blocks of natron (a carbonate of sodium) from their cargo, resulting in the formation of glass by the reaction of molten natron and the silica of the sand. However, production of glass under such conditions is incredible; the reaction involved is highly endothermic requiring a temperature never attainable in open air.[4] Thus, lacking dependable historical data about the origin of glass, we are bound to refer to present-day experience which favors one interpretation, namely that the glass-maker's formula was developed by the potter, as previously suggested. Additional evidence will be presented later on in this paper.

PROPAGATION OF THE GLASS INDUSTRY

Glass articles were exported from Egypt while their manufacture remained a strictly kept African secret until the domination of northern Africa by the Greeks of Alexander. Even the existence of glass made of sand was unknown to Aristotle, Alexander's contemporary, who understood glass to be a material altogether different from glass as we know it. Theophrastus, Aristotle's disciple, states[5] that he heard people claiming that there was a kind of glass made of sand. However, Plato had already distinguished[6] between the "many species of the genus glass" and the "many kinds of stones called cast," which must refer to articles cast from silicate glass. Herodotus, still earlier, mentions[7] cast-stone and gold earrings adorning tamed crocodiles in the vicinity of Lake Moeris and Thebes of Egypt. The accuracy of this information is attested by the finding of crocodile mummies with pierced ears (also by antique Egyptian glass earrings).[8]

With the advent of the Greeks light was slowly penetrating into the glass-maker's den. In the second century B. C., a lyric poet, Mesomedes, was given the opportunity to enter a shop and rhyme about the art

[1] Glazed bricks were used in the construction of Egyptian monuments (Temple of Belus, over 12,000 years old).

[2] POLYBIUS, Hist. 9.27.4.

[3] PLINY, Hist. Nat., 36.65.

[4] Also, lime cannot be made outside of a kiln or at least a covered pile although the temperatures of dissociation of the carbonates in the limestone are under ordinary pressures much lower than the temperature of glass formation. The case is different when certain kinds of stones, those used as fluxes for instance, are involved.

[5] LAPIDES 49.

[6] TIMAEUS 61b.

[7] Hist., 2.69.

[8] It is beyond doubt that both Aristotle and Theophrastus were aware of the existence of glass-cast stones; such were in use in their country since at least the era of the Mycenian civilization. They only doubted their nature and confused them with other African goods, known to them as glasses, which, as discussed further on, were also fashioned by a sort of molding process and served similar purposes.

of shaping the glass, a novelty to his world. While visiting, he followed "the workman powdering the glass, carrying it to the kiln, and putting the (loose) matter into the fire as (if it were) firm iron." Then he saw "the glass to melt like wax, burned by the all-devouring flames." He marveled watching "a trail flowing under the action of fire and the man trembling lest it should fall and burst, and finally putting a lump of it into folded claw tips."[9] The interpretation would not have been different if the verses were composed 2,000 years later, after a visit to a plant where glass is shaped by a kind of forceps. The poet had not visited a place where glass was manufactured by compacting earthy matter; the workman was dealing with raw material prepared elsewhere.

Obviously, the secret about shaping glass from a molten state emanated from Africa first, before the art of manufacturing it from sandy mixtures became known. However, knowledge of the nature and composition of the newly disclosed type of glass was spreading very slowly; Rufinus, another poet contemporary to Mesomedes, is not referring to this greenish, shiny and hard solid when comparing[10] the cheeks of a girl with glass. Still later an astronomer called [11] glass a substance dug from the ground. The necessity of proper sand for glass making is first mentioned[12] toward the beginning of the Christian era, almost simultaneously with the tale about Phoenicians and natron. It is believed, otherwise, that up to that time Alexandria in Egypt was the main exporting and, perhaps, manufacturing center. About two centuries later, Pausanias saw[13] flasks probably blown from silicate glass. Finally Byzantine Greece became the center of glass manufacture and through Venice, its dominion, the art was spread westward.

TREE AND KILN TEARS

Returning to the goods originally presented to the trade as glasses and known as such during the classical era, we find[14] that they were produced from special, easily workable stones, collectively called raw glasses. The latter were plentiful in Ethiopia where they were gathered or mined, and, along with murrhine stones, etc., were exported as products of Egypt. There were several multicolored and transparent kinds but they were all different from precious stones such as diamond or jacinth. This vague information is made clearer by Aristotle who enumerates the glasses[15] and also explains the process of their formation in nature in agreement with his conceptions about matter. Glasses were the gums, amber, frankincense, etc., as well as the exudates called tears and stalactites (tear-oozings from trees, resins). Some substances of mineral origin (fossil gums),[16] which could be softened by heat, were included. The glasses were found in many countries but Ethiopia was particularly renowned for its aromatic products, balms, resins, gums. As for their formation, Aristotle believed that the tree-tears become resins and further solidify by loss of liquid, just as "the clay hardens into brick and the stalactites in the caves become stones" when "by cooling they lose their innate heat and along with it their liquid." Likewise, the amber (found in water), supposed to be a tear of the black poplar, became solid when "its heat was driven away by the cold of the River" (ocean).[17] Such an explanation arises from observations as well as from practical experience; balsams and resins (oleo- or gum-), when properly heated, give a liquid distillate and a gum-hard residue.

Raw glasses could be shaped into commercial articles[18] (even combustible lenses) by "thickening" through heating and kneading.[19] There are gums cohesive to such a degree that even at ordinary temperatures their powders form compact lumps by mere pounding.

There came a time when the tears exuding in the potter's kiln and their many similarities with the tears of trees were observed. Both had solidified from the viscous state through exposure (causing cooling) and when reheated regained plasticity; both were glassy solids, translucent in thin slices, brittle, alike in conchoidal fracture, and, in powder form, of a similar light color; furthermore, the kiln-tears were shapable into forms hardly distinguishable from those made of gums. In conclusion the simple-minded ceramist became convinced that his peculiar product was another of the many kinds of tears, hence he named it "glass" too, despite the fact that it was decidedly heavier, hard, and of a singular color. The name was accepted by the trade, indicating that the difference in weight and in hardness did not matter; heavier articles, strings of beads, or other ornaments were appreciated just as well, if not more so, for their hardness. As for color peculiarity (kiln-glasses being dark, characteristically bluish-green), a new color was added to the glass hues. Another difference, the insolubility of the silicate products in oil, was of no interest to the public; only the varnish-maker was concerned about that and he knew what type of gums to select for his formulas. The newly introduced glass from which not only minor

[9] Anthologia Graeca 16.323, Loeb Classical Library. Our translation emphasizes the technical meaning of the poem.

[10] Anthol. Gr., 5.48.

[11] Achilles Tatius, 2.3.

[12] Strabo, 758.

[13] Perieg., 2.27.4.

[14] HERODOTUS, 3.24; DIODORUS III, 2.15; ARRIANUS, Peripl. M.R. 6; 17; 56.

[15] ARISTOTLE, Mete., 388b; 389a; Id., Hist. An., 5.22.5.

[16] This misled the scholars into considering glass as a kind of crystalline stone or rock crystal, or even porcelain.

[17] It is to be noted that all yellow gums often shared the name with the amber, known also as elektrons. Elektrons besides referred to other bright-yellow materials, like the gold-silver alloy of the ancients [AELIANUS, de Nat. An. 4.45; PAUSANIAS *op. cit.*, 5.12.16]. Today elektron signifies a magnesium alloy.

[18] Like the Amberoid, for instance, a present-day substitute for amber, compounded by compressing heated bits of amber, copal, etc. It is probable that the large and hollow pillar of the Hercules temple in Tyre, shining brightly by night [HERODOTUS 2.44], was a deceptively illuminated gum composition, the poor transparency of the gums making such an illumination possible.

[19] THEOPHRASTUS, *op. cit.*, 48, 49.

trinkets but also other goods of much greater usefulness could be shaped became eventually the more important and after a time remained the only kind known as glass.

The name of all these African goods, raw or finished, was heard and adopted in the Greek world as *hyelos* or *hyalos* (from which the combining forms hyalo- and hyal- in English are derived) and further west, as *glassa, glaesum* and the like.[20]

Many terms are derived from the names of the products at the place of their origin.[21]

A specific application of the gum-glasses was to preserve corpses, an Ethiopian and Egyptian practice[22] probably suggested by the discovery of lumps of gums with enclosures of visible and perfectly preserved insects (Aristotle uses the term animals). The origin of many human arts is doubtless due to the observation of natural phenomena, the causes of which are sufficiently understood to be duplicated. A corpse could be preserved in gummy matter and also be visible like the insect if, before application, gums could be softened to the consistency of balsams and then left to harden. This could be accomplished by mixing the gums with the previously mentioned distillate of resins—the liquid that, according to the ancient hypothesis, caused the balsams to remain soft before their gummification by mere loss of the same liquid.[23] Thus the application of a hard preservative coating became feasible, with only one difficulty still to overcome, namely impairment of the appearance of the naturally dark bodies[24] by the turbid coating. That must have necessitated the complete covering of the corpse with a gypsum enamel (using terra alba, a suitable kind of white earth) followed by painting with other pigments to resemble the deceased as closely as possible. All authors agree about this perfect preservation with visibility, even though the coated remains were left to outdoor exposure for a long time; however varied versions are given for the process of the glass application, due to the belief that gums can also be fluidified by mere heating without solvent addition. In fact, Herodotus deduces from reports of spies sent among the Long-Lived by the Persian king, who testified that a glass coffin—a "hollow pillar made of glass"—was set around the corpse. The source of information and the lack of details about the contact between corpse and glass raised comments from later authors,[25] not denying the fact, but doubting the feasibility of smearing bodies with molten gums without carbonization and marring of the appearance of the departed. The criticism is surely based on the understanding that the application of the glasses as practiced was not unlike the waxing for the same purpose by Persians[26] and Scythians, also mentioned by Herodotus.[27] But the conditions for possible application of either of these two different types of materials deviate widely. Pure beeswax softens between the fingers, becomes fluid at a temperature above 70°C. and, when molten, can incorporate hardening admixtures, like gums, to produce a desirable consistency and still retain the low melting point required. Gums, on the other hand, are brittle and, becoming fluid at a much higher temperature (around 300°C.), would not only darken, carbonize, and lose transparency by lengthy heating, but would also badly damage animal tissues in contact with them. Therefore, Herodotus does not scheme as accused by his critics; the gums, softened and compacted at low temperatures, could be safely applied to the corpse in the form of oily pastes or solutions, which harden to a glassy protective film, thickened by repeated coatings. Such oily compounds were widely used and for many purposes; such was the "cedar unguent,"[28] a mixture of gums and cedar-oil well

[20] The island Austravia in the "North Ocean" was renamed Glassaria by Caesar Germanicus because of the amber on its shores [PLINY *op. cit.*, 4.27 and 30; TACITUS, Germanicus 45].

[21] In Greek, *hyalos* is still the word for glass; the verb *hyalizo* means to shine and formerly meant also to be greenish like glass. Glass was originally and for thousands of years identified with its peculiar bluish-green color. The process of decolorizing was found long after the appearance of the product in the market. The Latins eventually differentiated the silicate from the other glasses by calling the former *vitrum*, being reminiscent of their green dye, the vitrum, extracted from the leaves of a brassicaceous herb of the same name, the woad (*Isatis tinctoria*). Consequently for purposes of distinction, the Greek *isatis* prevailed for the woad.

A precise use of the terms, however, was neither made by lay people, nor by authors. Thus, Pliny [*Op. cit.*, 35.27 (30)] uses "vitrum" for a component of the pigment "Anulare," from hearsay, since the pigment in question is never mentioned in his only reliable source of information, the classics; the pigment, he writes, was only recently introduced. It was compounded by mixing chalk and a powder of *"vitreis gemmis"* from orts of rings of commoners (*i. e.*, not of precious stones). Rings are specified (*anulis*), not stones set on rings, nor pieces of broken bracelets (*armillas*). Rings can be and are made of plastic materials, but not of glass so much readily breakable and far from safe for the fingers. The pigment besides, was used in painting to brighten the pictures of females (*muliebres picturae illuminantur*). Glass powder cannot impart any hue, no matter how applied, but light-colored paints can be formulated with the use of gums.

If another interpretation [BECKMANN, Hist. Inv. II 2600] for the use of the pigment be adopted, that of an item of ladies' make-up, we have as good an argument concerning its lack of hue and a sounder one against the use of powdered glass in contact with tender skins.

In favor of the former interpretation is the previously mentioned comparison of girls' cheeks with glass in the poem of Rufinus.

[22] HERODOTUS, 3.24; DIODORUS III., 2.15, 3.9 ET AL.

[23] Such a conception concerning the mechanism of hardening leads to a more precise interpretation of the natural hardening of the gums. Let the previously mentioned phrase of Aristotle about soft bodies that "by cooling lose their innate heat and along with it their liquid," be read "cool by loss of their liquid (unavoidable evaporation) and along with it their innate heat," in order to approach the real facts (chemical changes excluded) with consideration of the latent heat of evaporation also.

[24] Ethiopians of the Long-Lived race, perhaps Abyssinian people, and Egyptians of Meroe, obviously very dark colored, were the best known glass corpse preservers.

[25] DIODORUS, *loc. cit.*

[26] There is evidence, however, that also in Persia occasional gum preservation was applied, for example a tale [Pseudokallisthenes 2.18] about Alexander viewing the golden coffin containing the remains of Cyrus, "hair and all visible" under poured glass.

[27] 1.140; 4.71, 73.

[28] HERODOTUS, 2.87.

known in Egypt especially for its use in embalming;[29] another made with "tar-oil distillate" (turpentine); also the flagrant unguents of the Bible, etc.[30] The industries dealing with greases as raw materials were well developed in the African countries and produced highly esteemed products. Thus it is claimed that the steeping of flowers or generally odorous parts of plants in fats or oils to produce perfumes was there (perhaps in Egypt) originated. Also, there is a sound probability that fats were first saponified in areas watered by the Nile with its naturally alkaline waters. In Egypt a whole *Nome* (district) was named *Nitriotes* after natron or nitron, the sodium carbonate salt deposited and exploited locally.

The remarkable state of preservation of the elaborate coatings on coffins of mummies now in museums supports our suggestion. The coatings still shine bright, without any major cracks. Close examination reveals, besides fingerprints, casual marks as from the use of a knifelike tool with a wide edge for paste-smearing.

[29] DIOSCURIDES, *Mat. Med.*, 1.77.3.

[30] Apelles, the famous artist, used an unguent of his own secret formula for the protection of paintings against dirt and dust and for imparting gloss. Although not perfectly transparent it was invisible and detectable only by its smoothness to the touch [PLINY *op. cit.*, 35.36]. The paintings so protected were of the encaustic type.

VARNISHES

Mixtures of gums and vehicles were also called glasses until the public became familiar with silicate glass which overshadowed the gums and the gum-compounds in importance. Then these glasses from oils also lost their ancient name; they were labeled *veronikia* in reminiscence of the golden braids of Queen Veronike, wife of one of the Ptolemies Euergetes of Egypt, who sacrificed and dedicated her much-admired hair to the Goddess in evidence of conjugal devotion. Such was the brightness of these braids that upon their disappearance from the altar of the temple, the court astronomer reassured the infuriated king that the Goddess elevated them to the sky to shine perpetually as a constellation, the Coma Verenices.

From *veronikia* are derived the modern words for varnish in several languages. The varnishes were obviously light-colored, mostly yellowish, made with the use of yellow gums and ambers (elektrons), which were in turn renamed *veronikia* and so known to the Byzantine world as late as the twelfth century A.D.

MODIFIED HINSBERG PROCEDURE

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THE HINSBERG procedure for differentiating between primary, secondary, and tertiary amines, as presented in the standard laboratory manuals follows Hinsberg's general directions.[1] I have found the procedure generally unsatisfactory in the hands of the average student. The following process makes the differentiation more satisfactory and convincing.

It will be noted that no aqueous alkali is used in this method. The use of a large excess of the unknown amine and the use of alcohol as a solvent produces the amine hydrochloride on addition of the sulfonyl chloride, without the necessity of adding an aqueous KOH solution. The reaction is very rapid, and there is little danger of the formation of the ester as the time of contact with the alcohol is very short.

A relatively large excess of the solid or liquid amine to be tested—*i. e*, enough to fill the rounded portion of a 6-inch dry test tube—is dissolved in 3 ml. of 95 per cent ethyl alcohol. The alcoholic solution is treated with 3 drops of the sulfonyl chloride. The solution is then brought to a boil and again cooled under the tap. All sulfonyl chloride reacts and there is no subsequent need to hydrolyze any excess reagent.

The cooled alcoholic solution, consisting of excess free amine, amine hydrochloride, and sulfonamide, is now treated with excess of concentrated hydrochloric acid by adding the latter directly to the alcoholic solution. The substituted sulfonamide is usually precipitated as a white crystalline mass, or sometimes as an oil. All other by-products dissolve in the acidified solution.

The solid or oil is next filtered by gravity and then washed with warm water to remove excess acid and any possible solid amine hydrochloride. A hole is punched in the bottom of the filter paper and part of the precipitate is washed into another 6-inch test tube, using about 5 ml. of water in the process.

The suspended solid or oil is now treated with 4 pellets of solid sodium hydroxide and gently warmed. The material either dissolves or does not, and the student is satisfied with the result. The whole reaction is clean cut and convincing.

Tertiary amines fail to react to form the substituted sulfonamide in the first steps, and no precipitate appears on acidification. Again, the results are clean cut and convincing.

The method works except for the restrictions noted by Hinsberg in his original paper, but amine types mentioned by Bell[2] and Cheronis and Entrikin[3] have not been investigated.

[1] HINSBERG, O., *Ber.*, **23**, 2962 (1890).

[2] BELL, J., *Chem. Soc.*, 2787, **1929**.

[3] CHERONIS, N. D., AND J. B. ENTRIKIN, "Semimicro Qualitative Organic Analysis," Thomas Y. Crowell Co., New York **1947**, pp. 255–61.

"THE PIROTECHNIA" OF VANNOCCIO BIRINGUCCIO

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Vannoccio Biringuccio's "The Pirotechnia," written in the vernacular Italian of his native Siena, was published in Venice in 1540, a year or more after the author's death. It is the work of a practical mining engineer and metallurgical chemist. According to some who have written short accounts of his life, the book was dictated to a clerical in Rome during the last year of his life. The style would certainly suggest that this might be the case. He probably had no idea of writing a book until he realized that the end of his life was approaching. Like many before him he may have desired at least a small bit of earthly immortality. The author must have delighted in conversation. Throughout his busy and exciting life he gathered and remembered all sorts of information about the absorbing interests of his life; mining, the metal arts, ores, industrial chemicals of his time, and all that pertained thereto.

Before the publication of this book, which was the first printed book on the applied metal arts and the processes of ore reduction, the treatises dealing with the art of chemistry had been written by alchemists whose copious writings were clad in moralizing and mysticism. Knowledge of metallurgy and practical chemistry had been for the most part transmitted orally from teacher to apprentice. Trade secrets were zealously guarded and if a master craftsman did entrust his knowledge to the written form often the language would be purposely garbled and key words omitted.[1] There is little doubt that the knowledge of many processes and techniques was lost due to this haphazard method of propagation, and it was not until the invention of printing in the middle of the fifteenth century that a suitable medium was available.

Manuscripts had been copied by hand, and the first task of the printing press was to catch up on the backlog of works on philosophy and religion, the subjects in which most men of education were interested. Of the approximately 30,000 books which were printed in the fifteenth century few were concerned with science. Early in the sixteenth century some short works began to appear in Germany containing recipes in the chemical arts. One was the series appearing under the title "Probierbüchlein,"[2] the earliest being printed around 1510. There were also available several editions of the "Kunstbüchlien," dating from 1531. Some incunabular manuscripts, principally the "Schedula diversarum" of Theophilus, composed in the latter half of the eleventh century, and the elder Pliny's "Historia naturalis," contained material on chemical and metallurgical methods and were available at the time of Biringuccio. However, "The Pirotechnia" of Biringuccio was the first complete work concerning these subjects to be printed.

Biringuccio, baptized Vannoccio Vincenzio Austino Luca on October 20, 1480, was born in Siena of Paolo and Lucrezia di Bartolommeo Biringuccio.[3] Little is known of his early formative years. His father, an architect, and later the superintendent of streets in Siena, died when Biringuccio was 32 years of age. The fortunes of Biringuccio were intimately connected with those of the tyrannical Petrucci family which ruled his native Siena beginning in 1490 with Pandolfo and ending with the popular revolt of 1526.

Biringuccio worked in the mines around Siena and traveled widely during his life. At the age of 27 in 1507 he was in northern Italy after having traveled through Germany visiting mines and metal works. He was apparently under the patronage of Pandolfo on this trip and obtained support easily in subsequent travels. After Pandolfo died in 1512 his son, Borghese, succeeded to the rule and in the next year gave Biringuccio a a position in the Siena Armoury.

After the Petruccis were momentarily driven from Siena in 1515, Biringuccio, along with Borghese and Francesco Castori, the head of the mint, was accused of having debased the coinage alloy, and was declared a traitor to the Republic. It is interesting to note that in Book IX of "The Pirotechnia" the title of the third chapter reads, "Discourse and Advice on How to Operate a Mint Honestly and with Profit." During a short period of exile Biringuccio journeyed about Italy, until the reinstatement of the Petruccis in 1523 under Fabio, Pandolfo's younger son, by Pope Clement VII. Biringuccio's property and post at the Armoury were restored and his reputation must have been

[1] SINGER, C. J., "The Earliest Chemical Industry", The Folio Society, London, **1948**, p. 70.

[2] The American Institute of Mining and Metallurgical Engineers has recently published a translation of two of these small books. For comment on these translations see the review by HENRY M. LEICESTER, J. CHEM. EDUC., **27**, 233 (1950).

[3] (*a*) Biographical data were taken principally from the recent English translation of the third edition of "The Pirotechnia," by C. S. SMITH, and MARTHA T. GNUDI, The American Institute of Mining and Metallurgical Engineers, New York, **1942**, pp. ix–x. Other biographical sources are (*b*) "Encyclopedia Italiana," Instituto Giovanni Treccani, **1930**, Vol. VIII, p. 64; (*c*) U. THIEME, AND F. BECKER, "Allgemeines Lexikon der Bildenden Künstler," Verlag von Wilhelm Engelmann, Leipzig, **1910**, Vol. IV, p. 50; and (*d*) G. BUGGE, "Das Buch der grossen Chemiker," Verlag Chemie, G.m.b.H. Berlin, **1929**, Vol. I, pp. 70–84.

mounting rapidly at this time. If his personal assets were not considerable they must have been increased greatly a year later upon his being given a monopoly for the manufacture of saltpeter in the Sienese dominion. Saltpeter was a very important commodity not only for its use in making the large amounts of gunpowder necessary to supply a government in an almost constant state of warfare, or preparation for warfare, but also for its use in making the parting acid used to obtain gold from silver.

At the outbreak of the revolution of 1526 Biringuccio was in Florence but immediately returned to aid the deposed Petruccis in an attempt to retake Siena. The assault failed and the quest to regain control was abandoned completely when the Petruccis, including Biringuccio as director of artillery, and augmented by troops sent by the pope and troops from Florence, were defeated on July 25, 1526, in the battle of Porta Camollia. Biringuccio was again exiled from Siena and did not return until 1530 after the turbulent conditions had quieted down. During this second period of forced absence from Siena, Biringuccio traveled again through Germany, served for a while under Alphonso I d'Este, lord of Ferrara, Modena, and Reggio, and distinguished himself in the service of the Republic of Florence by casting the famous Lionfante cannon.

Upon his return to his native city Biringuccio once more became a man of property, being a Senator of Siena in January and February, 1531. He was appointed architect and director of the Opera del Duomo in 1535. His skill as an arms caster and fortress designer was exercised during the period 1531 to 1535 in the cities of Parma and Venice, and in the domain of Ercole d'Este. His fame brought recognition by Pope Paul III when he asked Biringuccio to come to Rome. Biringuccio assented and in 1538 at the age of 58 he became head of the papal foundry and director of papal artillery. He may have died in Rome soon afterward. Although no written record of his death has been found either in Rome or Siena, it is certain that he died before April 30, 1539, the date of a document in which certain debts due his heirs are discussed.

Biringuccio's "Pirotechnia" was the result of practical experience and knowledge gained by wide travel and intimate contact with his subject material. The book was certainly written from the standpoint of a practical man. It contains little discussion extraneous to the details of the subject and was written in the language of the common craftsman.

"The Pirotechnia" is always associated with the "De re metallica" of Georgius Agricola (1556). Both books deal with the same subjects. The great difference between them is a reflection of the training and life of the two authors. Biringuccio wrote from experience and from first hand knowledge, stating repeatedly in his book that he did not have any knowledge except that gained through his own eyes. He emphasizes practical methods throughout, *e. g.*, going to great lengths explaining the locating of a mine and how to work it properly in order to ensure financial success. He advocates the experimental approach upon the discovery of a certain ore. A variety of methods of smelting should be attempted until the proper conditions are discovered to give the best product. He appears to recognize his role in setting down the knowledge of technical processes when he states his purpose early in the book as follows:[4] "I have done this willingly in order that you may acquire more learning and because I am certain that new information always gives birth in men's minds to new discoveries and so to further information. Indeed I am certain that it is the key that arouses intelligent men and makes them, if they wish, arrive at certain conclusions that they could not have reached without such a foundation, or even nearly approached."

Besides his vast store of practical knowledge Biringuccio had other sources upon which he could have drawn. Throughout his book he acknowledges various early writers, among whom are Aristotle, Pliny, Theophilus, and Albertus Magnus. He gives credit to Agricola's "Bermannus" (1530) for certain material on silver ores. He may have been familiar with five editions of the "Probierbüchlein" and six editions of the "Kunstbüchlein," though these works were so sketchy in their descriptions that they were probably useless to him. Biringuccio probably knew the work of the alchemists well, taking great pleasure in deriding them from every angle after once praising them for the discovery of many practical methods. Biringuccio's literary style may leave something to be desired. His sentences were long and bulky and his paragraphing was done in random fashion. Much of the sentence structure and paragraphing was improved in later editions, and his style may be excused on the grounds that he was writing in ordinary language so that the practical man could understand him.

Agricola, on the other hand, was a scholar in every sense of the word, writing not only on metallurgy, mining, and the chemical arts, but also concerning medical, religious, philosophical, political, and historical subjects.[5] He wrote in Latin and his reputation was very well established at the time of the printing of "De re metallica." The work contained much the same material as "The Pirotechnia" with several additions and omissions, and appeared with much better, and more numerous, illustrations. The beautiful format of the house of Froben is another feature of "De re metallica" which gave it an advantage over the unattractive "Pirotechnia." Agricola gives one brief acknowledgment to Biringuccio's book, saying that by reading it he had refreshed his memory on certain processes he himself had observed while traveling through Italy. The fact is that Agricola copied *in extenso* certain parts of "The Pirotechnia," notably the portions on

[4] SMITH, C. S., AND MARTHA T. GNUDI, *op. cit.*, p. 28.

[5] AGRICOLA, GEORGIUS, "De re metallica," translated from the first Latin edition of 1556, by H. C. HOOVER, AND LOU H. HOOVER, The Mining Magazine, Salisbury House, London, 1912. A recent reprint has appeared in this country. Dover Publications, Inc., New York, 1950.

mercury and sulfur distillation, steel and glass making, and the purification of saltpeter, alum, salt, and vitriol by crystallization. He also expanded other portions by rearranging and adding details. As a consequence, "De re metallica" was a much more polished work, well arranged, and written in a fine literary style. For these reasons, it has overshadowed its predecessor, "The Pirotechnia," in the subsequent development of science. Being written by a true scholar it had much to do with other men of education soiling their hands with the ways of science.

Nevertheless, Biringuccio's book must have enjoyed great popularity in its day since four editions appeared in Venice before 1600. It probably had much use in the shops and foundries where the practical men and experimenters worked.[6] There has been a total of twelve editions—six in Italian, three in French,[7] one in Latin, one in German (1925), and the recent English translation. It has been suggested that there may be some significance to the fact that "De re metallica" appeared in a German edition as early as 1557 whereas no German translation of "The Pirotechnia" appeared until 1925.[8] The nationality of Agricola was, no doubt, a contributing reason for this. Also, the Germans were more active in the later development of modern science through their rigorous application of the experimental method. In the period before the middle of the nineteenth century the work of their compatriot, Agricola, satisfied their needs and their curiosity about the older period.

In any event, the references to "De re metallica" in scientific literature are much more numerous than those referring to "The Pirotechnia." Robert Hooke made a note in his diary on November 12, 1675, to look up Biringuccio's method for making steel. Some additional references have been recently pointed out.[9] There is a second reference in Hooke's diary dated March 1, 1677/8, in which Hooke mentions buying a copy of "The Pirotechnia" for two shillings. Further, Sir Thomas Browne in "Hydriotaphia" refers to Biringuccio's statement[10] concerning the long periods of time charcoal has been preserved underground (over 400 years).

"The Pirotechnia" is divided into ten books, the first four of which are particularly interesting to the chemist. These are entitled, respectively, Minerals and Their Smelting, Concerning the Semiminerals, Assaying of Metal Ores and Their Preparation for Smelting, and Concerning the Separation of Gold from Silver and the Bringing of Gold to its Final Perfection. Biringuccio makes a simple distinction between minerals and semiminerals, the former being ores from which useful metals can be smelted and the latter those materials which do not yield a metal. He takes the Aristotelian view of the constitution of matter from the four elements—fire, air, earth, and water—and has the alchemical conception of gold as the most perfect metal. Concerning the origin of gold he says that from the union of the elements "which are of equal force there is born a pleasing and perfect elemental mixture, and then after fermentation and decoction the elements finally become fixed, permanent, and joined together in such a union that they are almost inseparable, so that by the power of the heavens or of time or of the order of most wise Nature, or by all these together these substances are converted into that metallic body called gold."[11]

Because he did not use roasting processes, Biringuccio did not recognize the sulfide ores as minerals. He describes them as "mineral fumosities or the beginnings of minerals." Partly for this reason his list of metals includes only those which occur in the native state or as oxide ores. Among these are gold, silver, copper, lead, tin, and iron. He devotes much space to gold, silver, and copper, and their alloys. His interests are typical of Italy in the Renaissance. Trade during this period was growing. There was not nearly enough gold and silver in Europe to supply the actual needs of commerce. Hence Biringuccio's care in devising methods in processes of extraction so that the maximum yields of these important metals could be obtained.

He gives fairly good descriptions of the metals, including preparation, properties, and uses. Lead can be taken as an example. He says that the main importance of lead is in separating gold from mixtures containing silver and copper in the liquation process, which he is the first to describe. With respect to its origin Biringuccio states that lead has an overabundance of wateriness with its other substances in poor elemental mixture. It has the properties of liquefying easily and of leaving a stain when rubbed against an object.

He mentions calcination of lead alone or with other substances (tin, sulfur, common salt, or arsenic). When calcined alone he found that it gained in weight by 8–10 per cent, which is a fairly close approximation to the theoretical 7.7 per cent gain in weight when lead is converted to its oxide. The high value may be due to the formation of higher oxides. The idea that increase in weight was due to loss of lighter parts was compared with the increase in weight of an animal body after death. "For, as is evident, the spirits that sustain life are released and, since it is not possible to understand how these can be anything but substances with the qualities of air, the body remains without the aid of that which made it lighter by lifting it up toward the sky, and the heaviest part of the element has its natural force increased and is drawn toward the center. Thus

[6] The copy of the third edition in the Tulane Library looks as if it may have belonged to a practical man. It is well worn, contains several marginal notes in Italian, and, in the manner of the modern chemistry student's laboratory manual, has many pages marked with spilled substances.

[7] The first French edition (1556) is in the Tulane Library.

[8] READ, J., "Biringuccio in English," *Nature*, **151**, 569–70 (1943).

[9] MCKIE, D., "Three Historical Notes," *Nature*, **163**, 628 (1949).

[10] SMITH, C. S., AND MARTHA T. GNUDI, *op. cit.*, p. 177.

[11] *Ibid.*, p. 26.

the above difficulty is resolved by this explanation."[12] This idea was probably not original with Biringuccio and it has been suggested that it may have come from Leonardo da Vinci.[13] Neither did it stop with Biringuccio, being discussed by Jean Bodin in "Universae Naturae Theatrum" (1596) and by Galileo in "Il Saggiatore" (1623), among others.

One process in which Biringuccio apparently had no experience was brass making. After terming it "an alchemical art" he incorrectly described the calamine process, in which calamine (zinc carbonate or zinc oxide) is mixed with carbon, covered with copper, and heated. He omitted the carbon and packed the furnace with the calamine on top. The fact that zinc was the metal in calamine was not discovered until 1656 by Glauber. It appears that Biringuccio thought the copper itself had been changed in substance though he was familiar with the process of remelting the brass repeatedly until the copper was restored. He advised covering the furnace charge and also any brass to be melted with powdered glass "in order to prevent the color from evaporating."[14]

Biringuccio can be credited with the first description of the amalgamation process for separating metallic silver from its ores. Agricola does not mention the amalgamation of silver. In "The Pirotechnia" are given all the fundamental steps in the Patio process used by the Spaniards in Mexico, the discovery of which is usually attributed to Pedro Fernandez de Velasco in 1566.[15]

The semiminerals are classified in three groups by Biringuccio. The first contains the earthy materials which resemble rocks, are difficult to liquefy, and are best adapted for adorning paintings. Next are the watery metal-like materials which are easily liquefied and act like metals with fire. Among these he lists sulfur and antimony. The third group comprises the more watery materials which dissolve in water. In this class, along with vitriol, rock alum, and saltpeter, he includes quicksilver.

His descriptions of the semiminerals take the same form as those for the minerals. For example, he states that mercury is composed of viscous, subtle substance with an overabundance of wetness and coldness. It eats holes in metal vessels in which it is contained. The only metal which will not float on it is gold, which is drawn into it. For this reason it is useful in the extraction of gold since the gold can be obtained by filtering through a leather skin.

Sulfur, on the other hand, arises from a hot, unctuous, earthy substance, containing an excess of dryness and heat, and resembling fire in which it is easily fused. Because of these properties Biringuccio says that some believe sulfur to be the prime agent of Nature in making metals. Mercury lacks the warmness and dryness of the metals. Consequently, much effort was directed by the alchemists toward combining sulfur and mercury to yield the perfect elemental mixture, gold.

Biringuccio is probably the first to mention zaffre (cobalt oxide) and manganese (manganese dioxide). The principal use given was in the coloring of vitreous materials blue and purple, respectively. His description of the manufacture of parting acid by heating rock alum and saltpeter together, and its use in the purification of gold, is remarkable. He stresses the importance for commercial success of recovering the silver from the acid and even of recovering most of the acid and recycling it. He mentions twelve detailed precautions to be observed in parting. These are concerned with careful selection of the glass equipment, proper manipulation, recovery of all wastes, and suitable safety precautions.

Books V through VIII of "The Pirotechnia" are concerned with alloys, and the casting of bells and firearms. Book IX contains a variety of material on the chemical arts, ranging from the art of alchemy to the manufacture of lime and bricks. Finally, in Book X, directions for "making fireworks to be used in warfare and for festivals" are given. This is probably the section from which the short title of the book, "The Pirotechnia," is taken.

The over-all theme of "The Pirotechnia" is in keeping with an expression by Biringuccio of part of his philosophy. He says that he is sure that we understand that "of all things created by the most high God Himself or by Nature at His command, not one—even though it be an atom or the smallest worm—has been produced without some particular gift. And if we do not always discern this in everything, the cause lies in our defective vision, in our little knowledge, and in our lack of careful thought concerning the necessity of seeking hidden things."[16] This principle is ageless in its application and is still true today. Biringuccio has searched for hidden things with unusual success and his work bears the mark of an experimentalist throughout. He lived during a transition period in the development of science. He is one step ahead of the speculative alchemists in having eliminated the lizard tails and other superstitious constituents from various methods, and in having recognized the practical nature of many processes in their large scale development, but yet is only on the brink of the true explanations for most of the changes involved.

ACKNOWLEDGMENT

The author, a graduate student, would like to thank Professor Clara de Milt for suggesting the topic of this paper and for correcting the manuscript.

[12] *Ibid.*, p. 59.

[13] PARTINGTON, J. R., AND D. MCKIE, "Historical Studies on the Phlogiston Theory.—I. The Levity of Phlogiston," *Annals of Science*, 2, 363 (1937).

[14] SMITH, C. S., AND MARTHA T. GNUDI, *op. cit.*, p. 75.

[15] AGRICOLA, G., *op. cit.*, p. 297.

[16] SMITH, C. S., AND MARTHA T. GNUDI, *op. cit.*, p. 114.

A CONTINUOUS CLOUD CHAMBER

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SINCE the invention of the cloud chamber in 1911 by C. T. R. Wilson this instrument has become of immense importance in nuclear research and offers one of the most striking demonstrations of radioactivity. The need for a simple apparatus to demonstrate fog tracks due to radioactive particles has long been felt. Many simplified cloud chambers of the Wilson type have been described in which expansion and supersaturation of the moist air in the chamber is achieved by first squeezing and then releasing a rubber bulb connected to the chamber. Anyone who has ever tried to construct and operate one of these is aware of the many difficulties encountered.

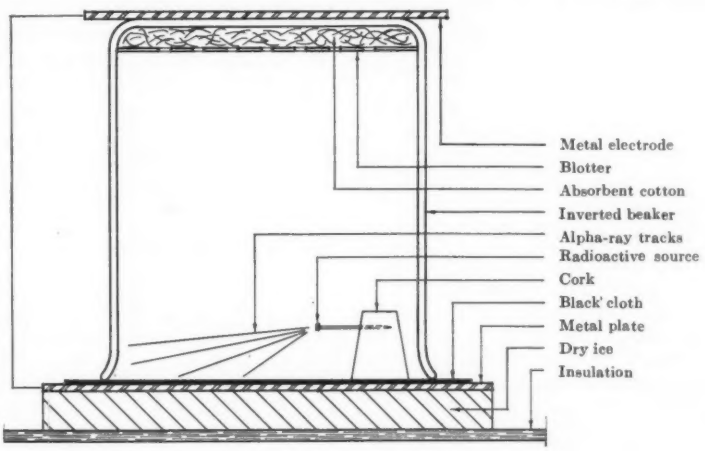
Langsdorf (*1*) and more recently Cowan (*2*), Needles and Nielsen (*3*), Miller, Fowler and Shutt (*4*) and Barnard and Atkinson (*5*) have described continuous, downward-diffusion cloud chambers in which supersaturation of the vapor in the chamber is achieved by cooling with dry ice. Some of the chambers described are of simple design, notably those of Cowan and of Needles and Nielsen. Similar cloud chambers, which were contained within crystallizing dishes and beakers, were set up by the author. Sometimes these gave excellent tracks and at other times refused to perform.

A study was then made of the many variables which could affect the performance of this type of cloud chamber. This work resulted in the design of a greatly simplified apparatus which has never failed to produce a striking display of alpha-ray and other tracks. Details of the construction of such a cloud chamber are shown in the figure.

So as to bring the chamber to a convenient eyelevel a cardboard carton is placed on a laboratory bench. Over this are spread several layers of cloth to insulate a layer of dry-ice chips, placed on top of the cloth. An aluminum, brass, or copper plate, 1 to 2 mm. in thickness, rests directly on the dry ice. The plate is covered with a piece of thin black cloth against which the tracks are viewed. A radioactive source mounted on a small cork is placed on the black cloth and then covered with an inverted beaker. Before inverting, a layer of cotton is placed in the bottom of the beaker and covered with a tightly fitting piece of blotting paper in which a number of holes have been cut. A piece of wire gauze may replace the blotter. The cotton is saturated with ethyl alcohol. A second metal plate is then placed on top of the inverted beaker, the upper and lower plates being connected through a reversing switch to a 220-volt direct-current power supply unit. The space just above the black cloth is illuminated with an intense horizontal light beam at right-angles to the alpha-ray tracks. The chamber is then swept free of dust particles and ions by applying the 220-volt current across the two plates. In less than a minute there is usually a beautiful display of tracks, which are sharpest just after the field has been reversed.

For continuous operation over a long period the chamber may be mounted directly on a block of dry ice several inches thick.

If a high voltage direct-current power supply is not available the chamber can be swept free of dust and ions most effectively by rubbing a piece of sheet plastic, such as cellulose acetate or Plexiglas, with a cotton cloth and bringing the charged sheet several times to the top of the chamber. The author has frequently observed the immediate appearance of tracks when the



charged sheet of plastic is laid on the inverted beaker. Periodically the plastic sheet must be recharged. Even a briskly rubbed sheet of paper will serve the purpose if the humidity is not too high. This method of removing ions is if anything, more effective than the 220-volt electric field. Sharper tracks are usually obtained. This is particularly true if a beaker larger than 400 ml. is used.

A number of different radioactive sources were tried. Polonium-tipped wires made especially for cloud chambers are available[1] as well as alpha-ray tips containing a small quantity of radium salt.[2] The radioactive-tipped needle from a spinthariscope gives good results. If none of these is available a small chip of luminous paint from a luminous-dialed watch or alarm clock cemented with sealing wax to the head of a pin makes a perfectly satisfactory source, particularly if the chip is first ignited on a crucible cover. The size of the chip and the amount of the radioactive material in it will of course determine the number of alpha-ray tracks obtained.

The simplified continuous downward-diffusion cloud chamber described in this paper was made possible when the following facts were accumulated:

(1) The temperature gradient between the solvent-evaporating surface and the cold metal plate is of importance. This may be varied by raising or lowering the solvent-evaporating surface, which, for good tracks, should not be more than about 10 cm. above the cold plate. The most spectacular tracks are obtained using a 600-ml. beaker, in which case a 5 cm. layer of cotton is placed in the bottom of the beaker so as to bring the solvent surface to within 10 cm. of the cold plate. A 250-ml. beaker requires only 0.5 cm. of cotton.

(2) Some means of sweeping dust and ions from the chamber is essential. If this is not done some tracks are often observable but they may not be very sharp, and usually disappear after a short time. Whether tracks are observed or not, ions are continually being formed by cosmic rays and by the radioactive particles ejected from the source. This results in a fine drizzle of condensed solvent. Only when the ions are swept out of the chamber can the required degree of supersaturation of the vapor be achieved, so as to produce a continuous display of fog tracks. If an electric field is used for this purpose a potential gradient of 20 volts/cm. is satisfactory.

(3) Many different solvents can be used but not all give good tracks. The results obtained with a number of different solvents are shown below.

Solvent used	*Result*
Absolute methanol	Good tracks
70% methanol	Good tracks
95% ethanol	Good tracks
Denatured alcohol	Good tracks
Normal propyl alcohol	Good tracks
Normal butyl alcohol	Very few faint tracks
Acetone	Very few faint tracks
70% acetone	Good tracks
Ether	No tracks
Pentane	No tracks

[1] Hammer Laboratories, La Mesa, California.
[2] Central Scientific Company.

(4) A black background against which to view the tracks is an essential part of the apparatus. A black cloth works very well, or the bottom metal plate may be blackened with flat black paint or stove polish.

(5) Good tracks were obtained with difficulty using a crystallizing dish set on dry ice and with a metal electrode and black cloth in the bottom of the dish, an apparatus similar to that described by Needles and Nielsen (3). That this was due to the low heat conductivity of the glass was shown by placing a sheet of glass between the dry ice and the metal plate in the apparatus as shown in the figure. Under such conditions a few tracks were observed only when the solvent surface was brought to within 4 cm. of the metal plate.

(6) Best results are obtained when the radioactive source is placed about 1 cm. above the black cloth. The region of adequate supersaturation of the vapor does not extend more than 1–2 cm. above the black cloth.

Other vessels may be used to replace the beaker in the figure. Extraordinarily beautiful and sharp tracks were observed in an inverted Petri dish. The inside bottom of the dish was lined with a piece of blotter in which a 4-cm. diameter circular hole had been cut. The blotter served to hold the solvent. As soon as a piece of charged plastic was brought to the surface of the Petri dish tracks became visible through the hole in the blotter. Such a setup has the advantage that the observer may look directly down at the tracks. Small inverted crystallizing dishes may be used in the same way.

Rutherford's experiment on the deflection of alpha-particles is readily demonstrated with a continuous cloud chamber. A lead sleeve is placed over the radioactive-tipped needle to obtain a beam of alpha-particles which are directed at a piece of gold foil mounted in front of the source. Most of the alpha-particles pass through the gold foil, indicating the open structure of the gold atoms. Many of them are deflected, as shown by their tracks, and on rare occasions tracks are observed moving backward from the gold foil. In the latter case the alpha-particle is assumed to have come very close to the nucleus of a gold atom, accounting for its large deflection.

Random cosmic-ray tracks are frequently seen in the cloud chamber and sometimes an alpha-ray track showing a sharp deflection representing a collision or near collision of an alpha-particle with an atomic nucleus.

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RADIOCOLLOIDS[1]

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IN 1912, while investigating methods for the separation of RaF(Po-210) from RaE(Bi-210) and RaD (Pb-210) in radiolead nitrate, Paneth (*1, 2*) found that when he dialyzed a solution of radiolead nitrate only the lead isotopes passed through the paper parchment or animal bladder membrane. This observation led to a large number of researches which established that radiotracers in minute concentrations (generally 10^{-9} *M* and below) may exhibit behavior similar to that of colloids (*3, 4, 5, 6, 7*). These substances have been termed radiocolloids.

Various investigators (*8*) have pointed out that radiocolloids exhibit the following properties: (*a*) diminished diffusion velocities, (*b*) stratification in the presence of gravitational or centrifugal fields, (*c*) coagulation or peptization by the addition of suitable electrolytes, (*d*) adsorption upon finely divided materials, filters, walls of containers, and other surfaces, (*e*) inability to dialyze through membranes permeable to ions, (*f*) discontinuous distribution of the radioactive material which is present as aggregates as evidenced by radioautographs, and (*g*) anomalous chemical behavior as evidenced by electrodepositions, nonequivalence of active and nonactive isotopes, and ion exchange phenomena.

Unlike ordinary colloids, the solubility product apparently does not have to be exceeded. The solubility product of $Pb(OH)_2$ is approximately 10^{-12}. Paneth (*3*) shows, however, that Pb(ThB) at a concentration of about 10^{-11} *M* in a 3.0×10^{-3} *N* ammonia solution is a radiocolloid. A simple calculation indicates that this cation concentration is far below that needed to satisfy the solubility product.

The following elements have been shown to form radiocolloids under proper conditions: Po (*4, 5*), Bi(*4, 5*), Pb (*4, 5*), Y (*9*), Zr(*8, 10*), Th (*11, 12*), Ba (*8*), La (*8*), Pu (*8*), Pa (*13*), Be (*14*), Sc (*15*), Nb (*8*), Mg (*16*), Sn (*17*), Ce (*18*), and Ti (*19*). The first three elements have been investigated quite extensively, while only a small amount of work has been done on the others. In most cases concerning these latter elements, only the recognition of radiocolloidal formation has been made.

DETECTION METHODS

A number of detection methods are available for the investigation of radiocolloids. Some of these are only qualitative, while others are semiquantitative. The most important of these methods will be treated in the following paragraphs.

Dialysis. This method, first used by Paneth (*1, 2*), is based on the idea that radiocolloidal aggregates will not pass through membranes permeable to ions. In practice, a membrane bag containing the radiotracer solution is suspended in a solution of identical chemical composition except for the tracer. The volume of the solution outside the bag is usually kept quite large as compared to that within the bag. The solutions in both compartments are stirred continuously throughout the time of dialysis. In most instances, at least 24 hours is required for equilibrium to be attained. Following the attainment of equilibrium, both solutions are sampled, analyzed for radioactivity, and a material balance is calculated so that absorption and adsoprtion by the membrane can be taken into account.

Filtration. Soddy (*20*) found that $Th(UX_1)$ could be separated on filter paper by filtering of a dilute solution of $UO_2(NO_3)_2$. Since that first filtration separation of a radiocolloid, several other investigators have used the method. In addition to filter paper, cotton and fritted glass filters have been employed. Schubert and Conn (*8*) have recently introduced ultrafiltration, using a modification of the apparatus of Greenberg and Greenberg (*21*). The filters that they used were "Visking" cellulose casing sacs.

Diffusion Coefficient Measurements. The first investigator to apply diffusion coefficient measurements to the investigation of radiocolloids was Hevesy (*22, 23, 24*). He found, as would be expected, that the diffusion coefficients of radioelements in the colloidal state were much smaller than those of the radioelements in the ionic form. Recent experiments (*8*) employing the techniques and apparatus of McBain and Liu (*25*) have been reported. The diffusion apparatus of Anderson and Saddington (*26*) should lend itself very readily to diffusion coefficient measurements of this type.

Ion Exchange. Schubert and Richter (*27, 28*) have shown that cation exchange resins present another method for the identification of radiocolloids. The uptake of tracer cations by an exchanger should decrease with an increase in the cationic concentration of the solution. However, should the tracer be in the radiocolloidal form, the uptake increases as the bulk cationic concentration is increased. This is attributed to the effects of the bulk electrolyte acting as a coagulant and the resin acting as an adsorbent.

An index for differentiating cation adsorption from colloidal adsorption by a cation exchanger may be

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expressed in terms of K, the equilibrium distribution coefficient, thusly

$$K = \frac{VT}{(100 - T)M}$$

where V is the volume of the solution, M is the mass of the cation exchange resin, and T is the per cent of the tracer adsorbed. When the tracer is present in the cationic form, K is constant as the ratio V/M is varied, K decreases when the bulk electrolyte concentration is increased, and K decreases in proportion to the concentration of an added complexing agent which forms an unadsorbed complex. When the tracer is present as a radiocolloid, K varies directly with changes in V/M, and K remains constant or increases as the bulk electrolyte concentration is increased. Adsorption of the radiocolloidal aggregates is also quite irreversible.

Centrifugation. In 1922, Lachs and Wertenstein (*29*) noted that the lower layers of a Po solution showed an increase in activity after standing for some time. The time required for this settling out was shortened many-fold by the centrifuge method originated by Harrington (*30*) for gases and later employed by Chamie and Guillot (*31*) for solutions. In practice, the solution is simply centrifuged for about an hour at a speed of 3000 to 6000 revolutions per minute in an ordinary centrifuge. After this, the solution is carefully removed, the centrifuge tube is washed out with concentrated acid to remove the precipitated radiocolloid, and the activities of the centrifugate and the wash solution are compared with the activity of the original solution.

Radioautography. The first use of photographic emulsions for the detection of radiocolloids was made by Chamie (*32*, *33*). She observed that several radioelements whether in the gaseous state, as amalgams, or in water solution showed discontinuities as evidenced by points of intense photographic activity. These points were interpreted as being colloidal aggregates. Several methods have been used to bring the radioactive solution into contact with the photographic emulsion (*6*). A drop of the solution may be placed directly on the emulsion, or the emulsion may be immersed in the solution. Alternately, a drop of the solution may be evaporated on a piece of mica, glass or other mounting material, allowed to dry, after which the mount is placed into contact with the photographic plate. Still another variation is to interpose a very thin cleavage of mica between the photographic plate and a drop of the solution. After exposure and development of the plate, microscopic examination will reveal the presence of colloidal aggregates in the original solution.

Starik and Diezenrot-Muisovskaya (*34*) claim that in the radioautographic method the aggregates are formed during the evaporation, and may not have been present in the original solution. Boyd (*35*) has shown recently that this objection is probably groundless. He has performed experiments showing that a drop of Po solution suddenly frozen on a photographic plate displays radiocolloidal aggregates similar to those obtained by the evaporation technique.

EXPERIMENTAL RESULTS

The section to follow will deal with experimental results obtained by various investigators. Many investigators have worked with mixtures of radioelements while others have been concerned with only one species in their researches. The subdivisions which follow are based upon the radioelements themselves, each being treated separately.

Polonium. The major nuclide that has been used in investigation of the radiocolloidal properties of Po is RaF(Po-210), and alpha-emitter with a half-life of 140 days. A few investigators have employed RaA(Po-218), an alpha-emitter having a half-life of 3.05 minutes. All other naturally occurring Po isotopes have half-lives of less than a second, and for this reason have not been satisfactory for such researches.

Dialysis. Paneth (*1*, *2*) in dialyzing solutions of RaD-E-F found that in acid solution the Po passed through the dialyzer whereas in basic and neutral media most of it was retained within the dialysis membrane. Starik (*36*) verified these results.

Electrophoresis. Godlewski (*37*, *38*, *39*) demonstrated that when a water solution of RaA-B-C was electrolyzed between Pt electrodes the RaA deposited on the anode. He assumed that the RaA was forming a negative hydrosol. Addition of HCl or $Al_2(SO_4)_3$ decreased the anodic deposition, while introduction of NH_4OH, KOH, or calcium citrate enhanced it. These results are in accord with the action that one would expect with ordinary colloids. When two different colloids are mixed in disproportionate amounts, the resulting aggregates take the sign of the one in excess. Using this fact, Godlewski added negative As_2S_3 and Pt colloids as well as positive Fe_2O_3 colloid to such solutions. In the former cases, the anodic deposition was enhanced, while in the latter it was diminished. Lachs (*40*), using Godlewski's method, found that RaA deposited on the anode in water and ethyl ether solutions, on the cathode in ethyl alcohol solutions, and on neither electrode in benzene and oil of turpentine solutions.

Both Paneth (*2*) and Haissinsky (*41*) found that RaD in acid solution goes to the cathode and in basic solution migrates to the anode.

Filtration. On adding $Al_2(SO_4)_3$ to water containing RaA-B-C, all three products were observed to remain on the filter paper when the solution was filtered. Godlewski, who observed this (*39*), also found that small quantities of HCl had the same effect but above an acid concentration of 1 N, the filter was inactive.

Centrifugation. The large majority of investigations on Po have been made with RaF employing the method of centrifugation.

1. Influence of Acids and Bases. Haissinsky (*42*), Guillot (*43*), Chamie and Haissinsky (*44*), Chamie and Korvezee (*45*, *46*), and Korvezee (*47*) have investigated the effects of acid and base concentrations on the

amount of Po that can be centrifuged in a given time. Curves of concentration versus per cent Po centrifuged have been reported for HCl, HNO_3 NaOH and NH_4OH. The curves for concentrations from 10^{-7} N to 10 N for these substances all seem to have about the same shape, which is that of a probability curve. The concentrations at which the greatest amount of Po is centrifugable for three substances are approximately 10^{-5} N for HNO_3 6×10^{-3} N for NaOH, and 10^{-1} N for NH_4OH.

2. Effects of Concentration. Chamie and Haissinsky (*44*) performed several experiments in HNO_3 solution to determine the effect of Po concentration on the per cent Po centrifuged. For an acid concentration of 5×10^{-2} N, the per cent Po centrifuged decreased as the Po concentration increased. Similar results were obtained in an acid concentration of 8×10^{-4} N. However, in 10^{-5} N HNO_3, 2.4×10^{-6} N HNO_3 and neutral solution, their results show at first a decrease and then an increase in the per cent Po centrifuged as the Po concentration increased.

3. Effects of Storage. Chamie and Haissinsky (*44*) have reported from experiments in 10^{-5} N HNO_3 that immediately after preparation 35 per cent, after a week 48 per cent, and after 45 days 71 per cent of the activity was removed by centrifugation.

The effects of using HNO_3 stored in glass for preparing Po solutions upon the per cent Po centrifuged was demonstrated by Rosenblum and Kaiser (*48*). They showed that more Po came down after a storage time of 8 days than did after one of 2 days, while Po solutions stored in paraffined containers showed no increase in removable Po after similar storage times.

4. Influence of Electrolytes. The effects of the presence of several electrolytes on the amount of Po centrifuged were studied by Chamie and Korvezee (*45*). Using 0.1 N solutions, the per cent Po centrifuged increased through the series: $NaNO_3$, $La(NO_3)_3$, $Ca(NO_3)_2$, $Pb(NO_3)_2$, and $AgNO_3$. Later Korvezee (*47*) studied the influence of varying the concentration of the added electrolyte. He employed solutions containing $AgNO_3$, $NaNO_3$, $Ba(NO_3)_2$, $TlNO_3$, $Pb(NO_3)_2$, and Na_2SO_4. In every case, a curve similar to the probability curve was obtained when the per cent Po centrifuged was plotted against the electrolyte concentration. The maxima in the per cent Po centrifuged occurred at the following approximate concentrations: 0.04 N in $AgNO_3$, 0.04 N in $NaNO_3$, 0.1 N in $Ba(NO_3)_2$, and 0.047 N in $TlNO_3$, 0.035 N in $Pb(NO_3)_2$, and 0.041 N in Na_2SO4.

5. Miscellaneous Experiments. Guillot (*43*, *49*) reported that after saturating a solution 1 N in HCl and containing 2.25×10^{-11} g. of Po per milliliter with H_2S 97.5 per cent of Po could be centrifuged. Haissinsky (*50*) has observed that a 1 N tartaric acid solution containing 7.7×10^{-9} gram atoms of Po per liter retained practically all its activity after a prolonged centrifugation. However, upon making the solution basic with NH_4OH, 42 to 100 per cent of the activity was removed by centrifugation. Haissinsky (*41*) also has shown that the addition of pyrogallol to 0.1 N nitric and acetic acid solutions of Po allows 98.3 and 72.6 per cent of the activity to be centrifuged out.

6. Action of Gravity. In a sense the action of gravity may be considered as centrifugation. Noting that the activities of Po solutions that had been allowed to stand for some time seemed to be greater in lower levels than near the surface, Lachs and Wertenstein (*29*) ran a series of experiments in burets by withdrawing samples from the bottom and from the surface. After standing for 24 to 48 hours, they observed a lower/upper activity ratio much greater than 1 in neutral and 0.1 N NH_4OH solutions and a ratio only slightly greater than 1 in a 0.04 N solution. Desiring to investigate the distribution more precisely, they carried out a second series of experiments in a vertical glass tube which had five capillary tubes located 2.4, 18.5, 34, 50, and 54 cm. below the liquid surface which could be used for withdrawing samples. Their results on neutral solutions show an increase of activity in lower portions even after one-half an hour of standing Starik (*36*) reported similar experiments using two tubes, one containing an acid solution, the other an alkaline solution. He was unable to verify the results of Lachs and Wertenstein.

Diffusion. Hevesy (*23*, *24*) and Paneth (*2*) investigated the diffusion constants of Po in various media. They obtained the following values: 0.77 $cm.^2$/day in 0.1 N HCl, 0.31 $cm.^2$/day in 0.001 N HCl, and 0.19 $cm.^2$/day in 0.5 N NH_4OH. The decrease in diffusion coefficients was interpreted as an indication of an increase in the average size of the diffusing particles.

Radioautographs. Chamie (*32*, *33*, *51*, *52*) reported that the presence of aggregates of Po in neutral, ammoniacal, and weak HCl solutions could be detected by the photographic method. Chamie and Marques (*52*) found that the per cent Po deposited as colloidal aggregates from solutions strongly acid in HNO_3 is a function of the concentration of the Po. A maximum per cent deposition is observed at approximately 3.4×10^{-10} g. of Po. On either side of this quantity the per cent deposited decreases.

Blau and Rona (*53*) made further use of the photographic method, reporting a large number of observations. Numerous aggregates were observed in solutions of Po in 0.1 N HNO_3, 1 N chromic acid, and 1 N HCl saturated with H_2S. Some aggregates were noted in KOH. 10^{-4} N HNO_3 plus tartaric acid, and hydrazine in HCl solutions as well as in glycerin and sulfuric acid. Only slight aggregate formation occurred in 1 N HNO_3 while no aggregates were detected in 4 N HCl saturated with H_2S and in 1 N HCl. Deposits of Po on electrodes were also investigated by these workers using radioautographs. Aggregates appeared on the electrodes when deposition was made from 10^{-4} N HNO_3 and chromic acid solutions, while no aggregates could be detected on the electrodes when 1 N HNO_3 and H_2SO_4 solutions were used.

Bouissieres, Chastel, and Vigneron (*54*) employed the method to determine the states of Po in

H_2O, C_2H_5OH, and $(CH_3)_2CO$ solutions. In H_2O aggregates were detected, while in the other two solvents only a very few aggregates could be detected. They also observed that the number of aggregates increases markedly after the solution has aged several hours. Starik (*36*) found aggregate formation in acid, neutral, and alkaline solutions of Po. Boyd (*35*) has recently reported that a radioautograph of a drop of a Po solution suddenly frozen shows a colloidal type of aggregation.

Adsorption. Leng (*55*) and Starik (*36*) have carried out a number of experiments to determine the adsorption of Po by glass, parchment, colloids, and dust. These experiments point out that the adsorption is almost always greater in acid solution than it is in alkaline.

Bismuth. A number of naturally occurring Bi isotopes have been used in the investigations of the radiocolloidal properties of this element. These include RaC(Bi-214), RaE(Bi-210), and ThC(Bi-212). In addition, Bi-204 and Bi-206 have been shown to exist in the radiocolloidal form.

Dialysis. In performing dialysis experiments on RaD-E-F solutions, Paneth (*1, 2*) observed that RaE passed through the membrane when acid solutions were employed, but did not pass through when neutral or basic solutions were utilized.

Electrophoresis. Godlewski (*37, 38, 39*) showed that a solution of RaA-B-C when electrolyzed between Pt electrodes produced a deposit of RaC on both the anode and the cathode. Lachs (*40*) observed only an anodic deposit in water, and found that in C_2H_5OH RaC deposits on the anode, while in $(C_2H_5)_2O$, C_6H_6, ethyl malonate, and oil of turpentine it appears on the cathode.

Godlewski (*38, 39*) also demonstrated that the addition of HCl, $Al_2(SO_4)_3$, or a positive colloid such as Fe_2O_3 caused a decrease in the amount of RaC deposited at the anode. The addition of NH_4OH, KOH, calcium citrate, or negative colloids such as As_2S_3 and Pt caused the anodic activity to increase As in the case of Po, he interpreted these results as being evidence for colloidal RaC.

Filtration. The report has been made by Godlewski (*39*) that RaC may be filtered from a solution of Rn-222 decay products using filter paper. The addition of HCl in concentrations above 1 *N* causes the filter paper to be inactive. Lachs and Herzfinkel (*56*) have carried out similar experiments verifying this work. Gile, Garrison, and Hamilton (*57*) were able to separate Bi-204 and Bi-206 from a lead target by dissolving it in 16 *N* HNO_3, evaporating to dryness, dissolving in excess 10 per cent NaOH, diluting with an equal volume of H_2O, and filtering through two consecutive filter papers. The filters retained over 98 per cent of the Bi activity.

Centrifugation. Herzfinkel and Wroncberg (*58*) showed that RaC could be brought down by centrifugation of Rn-222 decay products dissolved in the following solvents: H_2O, C_2H_5OH, $(CH_3)_2CO$, and C_6H_6.

Werner (*59, 60*) showed that the amount of ThC centrifuged from solutions of ThB-C prepared from purified (by previous centrifugation or ultrafiltration) water was less than that centrifuged from such solutions prepared from nonpurified water. Further experiments by Werner indicated the influence of acids, bases and salts upon the amount of ThC that could be removed by centrifugation. Using HCl, HNO_3, H_2SO_4, NaOH, NH_4OH, NaCl, K_3AsO_4, $La(NO_3)_3$, and sodium citrate, he plotted curves of per cent Bi centrifuged versus the logarithm of the electrolyte concentration. The acid curves exhibited maxima at about 10^{-3} *N* in the range 10^{-7} to 10^{-1} *N*. The curves for the bases showed maxima at about 10^{-2} *N* in the range from 10^{-3} to 10^{-1} *N*. The citrate data showed a steady decrease in centrifugable Bi as the citrate concentration increased, while the remaining salts gave lines which were essentially parallel to the concentration axis.

Lachs and Wertenstein (*29*) found that the action of gravity on RaD-E solutions caused a concentration of the activity in the lower layers after 30 hours standing.

Diffusion. Paneth (*2*) determined the diffusion constant of RaE in 0.001 *N* HCl to be 0.45 $cm.^2$/day. He also states that the diffusion constant in alkaline solution is less, but exact values are not given.

Radioautographs. Hahn and Werner (*61*) employed the photographic method to detect aggregate formation of ThC in various media. They found many groups in neutral 0.01 *N* HCl, and Na_2SO_4 solutions, a few groups in 0.033 *N* HCl, and almost no groups in 0.1 *N* HCl, NaCl, and mannite solutions.

Adsorption. King and Romer (*62*), on the basis of their work concerning the adsorption of ThC on the walls of glass vessels and the work of Leng (*55*) dealing with the adsorption of ThC on glass and paraffin, state that there is a maximum adsorption at a pH of from 5 to 7 and that this adsorption drops off rapidly at a pH of from 2 to 3.

Electrodeposition. Hevesy (*63*) found that the per cent ThC deposited on Cd and Zn plates which were dipped in ThB-C solutions increased as the acidity in HNO_3 increased from 0 to 1 *N*.

Lead. Experimental investigations of lead have utilized RaD(Pb-210), ThB(Pb-212), and RaB-(Pb-214).

Dialysis. Paneth (*2*) reported that the dialyzability of ThB is greatly reduced in ammoniacal solution. Leng (*55*) reported that the dialyzability of ThB in a 10^{-3} *N* KOH solution was less than that of ThB in a neutral solution.

Electrophoresis. Paneth (*2*) showed that ThB in in both dilute HCl and dilute NH_4OH gathers in the cathode compartment when a membrane is placed over the entrance to the compartment.

Godlewski (*37, 38, 39*), in the electrolysis of Rn-222 decay product solutions between Pt electrodes, observed that RaB deposited on the cathode. The addition of HCl or $Al_2(SO_4)_3$ increases the activity of RaB on the cathode, whereas negatively colloidal As_2S_3 or Pt decreases the cathodic activity.

Filtration. On filtering a solution of RaA-B-C through filter paper, Godlewski (*39*) found the filter to be active wih RaB. Upon adding HCl to the solution, RaB no longer was filterable. Lachs and Herzfinkel (*56*) verified these observations.

Centrifugation. Werner (*59, 60*) centrifuged solutions of ThB-C containing varying concentrations of a number of electrolytes. The addition of HCl, HNO_3, or H_2SO_4 in concentrations from 10^{-5} *N* to 10^{-1} *N* caused the per cent ThB centrifuged to decrease as the amount of acid was increased. In all cases, no ThB could be precipitated at 10^{-1} *N* acid. The addition of NaOH or NH_4OH caused an increase in per cent centrifugable ThB up to 10^{-2} *N* after which the per cent decreased. The addition of NaCl, $La(NO_3)_3$ or sodium citrate caused a decrease in the centrifuged ThB as the salt concentration increased from 10^{-4} *N* to 10^{-2} *N*.

Diffusion. Hevesy (*23, 24*) reports that the diffusion coefficient of ThB in 0.1 *N* HCl is 0.67 *cm.*^{2}/day, in neutral solution is 0.68 *cm*^{2}/day, and in 0.1 *N* NH_4OH is 0.37 *cm.*^{2}/day.

Adsorption. King and Romer (*62*) report on the basis of their researches and those of Leng (*55*) that for ThB there is a maximum adsorption on glass at a pH of about 9 to 10 and that there is a sharp dropping-off of adsorption at a pH of about 7 to 8.

Electrodeposition. Hevesy (*63*) noted that when a Cd sheet was dropped into a solution of ThB-C, the ThB deposited on it increased as the nitric acid concentration increased from 0 to 1 *N*.

Thorium. Soddy (*20*) noted that UX_1 deposits almost quantitatively on filter paper when a solution of $UO_2(NO_3)_2$ is filtered. Kurbatov (*11*) made a study of the radiocolloidal properties of UX_1 as to the effects of pH, foreign anions, nitrate and carbonate, and the foreign cation, uranyl ion, on the amount of UX_1 removable by filtration. They show that the amount of UX_1 removable from 10^{-13} *M* solutions at different pH values and with varying concentrations of foreign ions is very similar to the behavior of 10^{-4} *M* solutions of Th. With the amount of carbonate in solution minimized, an increase in pH from 1.12 to 6.76 caused an increase from 10 to 98 per cent in the amount of UX_1 removed. Ammonium hydroxide was used to adjust the pH values. As the nitrate ion concentration was decreased, the per cent removable UX_1 increased. The effect of bicarbonate ion was shown to be opposite that of an increase in pH. As the uranyl ion concentration is increased, the UX_1 removed is decreased.

Kurbatov and Silverstein (*12*) determined the coagulator isotherm of Th at a pH of 3.5 using UX_1 as a tracer. They found that as the total Th increased from 10^{-9} gram-atoms per liter to 10^{-5} gram-atoms per liter, the per cent Th coagulated during a period of 2 hours decreased from 82.3 to 13.0 per cent.

Booth (*64*) reported that UX_1 is quantitatively adsorbed from aqueous solutions of pH greater than 5.0 by the walls of a glass beaker when heated to boiling for an hour.

Yttrium. Yttrium-86 was employed by Kurbatov and Kurbatov (*9*) to make studies of the radiocolloidal properties of this element. They employed the method of filtration and investigated the effects of pH, NH_4Cl, and Y concentration on the per cent Y filtered. As the pH was increased from 3.8 to 8.8 in a 10^{-11} gram-atom per liter Y solution with the NH_4Cl concentration constant, the per cent filterable Y increased from 13.6 to 100.0 per cent. Studies of larger quantities of Y in solution employing Y-86 as a tracer showed similar behavior. At a pH of 7 the per cent Y filtered from a solution in the concentration range 3×10^{-2} to 10^{-5} gram per liter was found to increase with a decrease in concentration. At a concentration of about 10^{-5} gram per liter, the per cent filtered Y coincides with the per cent Y removed from minute concentration solutions. As the concentration of NH_4Cl is increased in both 0.159 milligram per liter and 10^{-11} gram per liter Y solutions, the per cent Y filtered decreases.

Zirconium. Kurbatov and Kurbatov (*10*) studied the filtration of Zr from its solutions both in dilute and minute concentrations using Zr-89 as a tracer. In the concentration range 1.5×10^{-4} to 1.5×10^{-11} gram-atom per liter, the fraction filtered increased with dilution to 2.7×10^{-6} gram-atom per liter. At 2.7×10^{-6} gram-atom per liter and lower, the filtered fraction reaches a maximum and remains constant. Added NaCl increases the filterable Zr above 10^{-5} gram-atom per liter, but below 10^{-6} gram-atom per liter, this effect is reversed. An increase in pH results in an increase in the filtered Zr at all concentrations in the range studied.

In a solution of $UO_2(NO_3)_2$ at its natural pH containing most of the fission products, Schubert and Conn (*8*) found that Zr-95 did not dialyze completely. They then prepared 0.48 *M* solutions of $UO_2(NO_3)_2$ which were made 0.5 *M* in various electrolytes. The dialyzed Zr in these solutions was 15 per cent with $NaNO_3$, 18 per cent with Na_2SO_4, 30 per cent with no added electrolyte, 66 per cent with HNO_3, and 100 per cent with H_2SO_4, conditions for all experiments being the same. In another series of experiments, 25 per cent of the Zr dialyzed in 0.01 *M* HCl, 47 per cent in 0.1 *M* H_3PO_4 or 0.1 *M* $H_2C_2O_4$, and 100 per cent in 0.1 *M* H_2SiF_4.

These same investigators also observed that in ion exchange the per cent Zr adsorption increased as the concentration of $UO_2(NO_3)_2$ increased, this action being characteristic of a colloidal type of adsorption. Schubert and Richter (*28*) have shown the effects of complexing agents and acids upon the ion exchange adsorption of Zr. The diffusion coefficients of Zr under various conditions were also measured (*8*). In 0.48 *M* $UO_2(NO_3)_2$ the diffusion coefficient is 0.22 *cm.*^{2}/day, in 1.2 *M* $UO_2(NO_3)_2$ 0.13 *cm.*^{2}/day, in a solution 0.48 *M* in $UO_2(NO_3)_2$ and 0.5 *M* in H_2SO_4 0.81 *cm.*^{2}/day.

Niobium. Schubert and Conn (*8*) performed experiments on the radiocolloidal properties of Nb along with their studies on Zr. The radioactivity of Nb-95 was used to trace the behavior of the element. In 0.48 *M* $UO_2(NO_3)_2$ solutions which had been made 0.5

M in several electrolytes, they detected the following per cents Nb dialyzed in a given hydrolysis time: 22 per cent with no added electrolyte or $NaNO_3$, 74 per cent with Na_2SO_4, 77 per cent with HNO_3, and 80 per cent with H_2SO_4. In another set of experiments, 21 per cent of the Nb dialyzed in 0.01 *M* HCl, 74 per cent in 1.0 *M* HNO_3 or 0.1 *M* H_3PO_4, 80 per cent in 0.1 *M* $H_2C_2O_4$, and 100 per cent in 0.1 *M* H_2SiF_6.

Much the same as in the case of Zr, Nb adsorption in ion exchange experiments behaved as if Nb were a colloid. Experiments by Schubert and Richter (*28*) show the effects of complexing agents on the ion exchange adsorption of Nb. The diffusion coefficients of Nb in $UO_2(NO_3)_2$ solutions have been reported by Schubert and Conn (*8*) as 0.22 *cm.*^{2}/day in 0.48 *M* $UO_2(NO_3)_2$, 0.11 *cm.*^{2}/day in 1.2 *M* $UO_2(NO_3)_2$, and 0.21 *cm.*^{2}/day in solutions 0.48 *M* in $UO_2(NO_3)_2$ and 0.5 *M* in H_2SO_4.

Lanthanum. The dialysis of minute concentration solutions of La-140 in fission products has been carried out by Schubert and Conn (*8*). They find that 100 per cent of the La is dialyzable in water solutions, in 0.01 *M* HCl or in 1.0 *M* HNO_3, 93 per cent in 0.1 *M* H_3PO_4, 66 per cent in 0.1 *M* H_2SiF_6, and 26 per cent in 0.1 *M* $H_2C_2O_4$.

Diffusion coefficients of La-140 were reported by these same authors. In 0.48 *M* $UO_2(NO_3)_2$ the diffusion coefficient is 0.35 *cm.*^{2}/day, in 1.2 *M* $UO_2(NO_3)_2$ 0.23 *cm.*^{2}/day, in solutions 0.48 *M* in $UO_2(NO_3)_2$ and 0.5 *M* in HNO_3 about 0.5 *cm.*^{2}/day, in solutions 0.48 *M* in $UO_2(NO_3)_2$ and 0.5 *M* in H_2SO_4 0.31 *cm.*^{2}/day, in 0.235 *M* HNO_3 0.53 *cm.*^{2}/day, in 1.2 *M* HNO_3 0.51 *cm.*^{2}/day, and in 5.0 *M* HNO_3 0.45 *cm.*^{2}/day.

Other Elements. King (*65*) demonstrated that centrifugation of mixtures of Np and Pu in very dilute HNO_3 solution brought about a precipitation of activity this activity being almost entirely due to the Pu. He also reports that at a pH of 3 Pu is adsorbed on glass, but Np is not. As the concentration of acid in solution increases, the amount of Pu adsorbed is decreased. Dialysis experiments (*8*) performed with human urine containing Pu show at a pH of 6.2, 52 per cent of the Pu dialyzes, while at a pH of 2.1, 80 per cent dialyzes. Only a small fraction of Pu is ultrafilterable from human blood or plasma (*66*).

Maddock and Miles (*13*) centrifuged a series of Pa-233 solutions of different compositions. Their results definitely indicate that Pa was being precipitated, but for the most part were too erratic to establish trends. However, they were able to show that as the concentration of HNO_3 increased from 1.0 *N* to 6.0 *N* and of HCl increased from 0.5 *N* to 6.0 *N* the per cent of Pa centrifuged decreased.

Haymond, Bowers, Garrison, and Hamilton (*16*) found that carrier-free Mg-27 in alkaline solutions forms radiocolloidal aggregates which may be removed by filtration.

Gile, Garrison, and Hamilton (*15*) described a method for separating carrier-free Sc-44, 46, 47, 48 as a radiocolloid from an NH_4OH-H_2O_2 solution by filtration.

Maxwell, Haymond, Bomberger, Garrison, and Hamilton (*17*) reported that carrier free Sn-113 collected in HNO_3 or H_2SO_4 forms a radiocolloid and is adsorbed onto the walls of the containing vessel.

Allen, Pool, Kurbatov, and Quill (*19*) reported that Ti-45 in concentrations as low as 10^{-15} g. per liter could be filtered out of Sc solutions made basic with excess NH_4OH.

Kurbatov and Pool (*18*) separated Ce-139 from solutions of activated La by coagulation of the Ce at a pH of 3.1 to 3.3 in the presence of MnO_4^- and NO_3^- ions. The Ce was then freed of Mn by a second coagulation at a higher pH after the NO_3^- ion had been replaced by the Cl^- ion.

Haymond, Garrison, and Hamilton (*14*) showed that Be-7 could be separated as a radiocolloid from Li solutions containing it. At a pH of 9, obtained by the addition of NH_4OH, more than 90 per cent of the Be could be removed by filtration through a glass filter disc.

Schubert and Conn (*8*) recorded diffusion coefficients for Ba-140 in various media. In 0.48 *M* $UO_2(NO_3)_2$ they gave a value of 0.37 *cm.*^{2}/day, in 1.2 *M* $UO_2(NO_3)_2$ 0.25 *cm.*^{2}/day, in a solution 0.48 *M* in $UO_2(NO_3)_2$ and 0.5 *M* in HNO_3 0.47 *cm.*^{2}/day, in a solution 0.48 *M* in $UO_2(NO_3)_2$ and 0.5 *M* in H_2SO_4 0.15 *cm.*^{2}/day. They explain the low value in H_2SO_4 solution as being due to radiocolloidal formation brought about by the insolubility of $BaSO_4$.

THEORY

The method of formation and the nature of the radiocolloidal aggregates in solutions have been of outstanding interest to almost all investigators in this field. Arising almost simultaneously with Paneth's discovery (*1*, *2*) of radiocolloids, two schools of thought developed as to the origin and nature of these formations. One group adopted the view, originated by Paneth, that the radioelements formed true colloids. The other group, believing the idea first suggested by Zsigmondy (*67*), took the view that the radioactive ions are adsorbed by impurities in the solution, thus simulating colloids. More recent investigators (*5*) seem to believe that an acceptance of both actions is necessary to explain radiocolloids.

The True Colloid Theory. Factors which favor the true colloid theory may be summarized as follows:

1. Careful purification of solvents before using them to prepare solutions of radioelements diminishes radiocolloidal formation, but does not eliminate it (*47*, *58*, *59*).

2. Hydrogen ion concentration conditions most favorable to the formation of radiocolloids do not correspond with those most favorable to adsorption on various bodies (*36*, *55*, *62*, *68*).

3. Variation in the per cent of radiocolloid centrifuged with an increase in the radioelement concentration indicates that another phenomenon in addition

to adsorption is involved in the radiocolloidal state. Chamie and Haissinsky (*44*) showed the influence of the concentration of Po on the per cent Po centrifuged from HNO_3 solutions of varying acidities. If the pH is of the order of 4 or less the per cent Po removed decreases with an increase in Po concentration, but in solutions nearing neutrality the per cent centrifuged increases with Po concentration. For true colloids, one would expect the per cent centrifuged to increase with the concentration due to enlargement of the particles, while with adsorption, one would expect the reverse since equal volumes of solution should contain practically the same quantity of impurities. To explain their results, Chamie and Haissinsky accepted two superposable phenomena stating: (first) that when the acid concentration is relatively large or the radioelement concentration is relatively small, adsorption is the important factor, and (second) that when the acid concentration is relatively small and the radioelement concentration relatively large, the true colloid phenomenon predominates. Kurbatov, Kurbatov and Silverstein, (*9, 10, 11*) have shown that as the amount of Th, Y, or Zr increases in a solution the per cent filterable decreases.

4. The intentional addition of impurities capable of acting as adsorption centers will not eliminate the ionic properties of ionic solutions of radioelements nor enhance the properties of radiocolloids in solutions (*5, 31*).

5. Variation of radiocolloidal properties as a function of pH or concentration of salts follows a course very similar to that observed for macroscopic hydrosols of the corresponding inactive isotope. Haissinsky (*69*) has shown that the influence of pH and salts on the per cent ThC centrifugable is analogous to the influence of the same factors on the amount of Bi hydrosol in solutions containing macroscopic amounts of Bi. He states that since impurities are not important in the latter case, and since the identity of the phenomena in both cases is apparent, then impurities are not important in the ThC instance. Kurbatov (*11*) has shown that the coagulation of Th from 10^{-4} *M* solution at different pH values and foreign ion concentrations is similar to the coagulation of UX_1 from 10^{-13} *M* solutions. Kurbatov and Kurbatov (*9, 10*) report similar data for Y and Zr.

6. Radioautographs of radioelements adhering to inactive impurities should have a clear center and a diameter greater than twice the range of the radioactive particle, while radioautographs of true colloids should have black centers and diameters equal to twice the range of the radioactive particles. Both types have been observed, (*6, 70, 71*).

The Impurity Theory. The following summary presents the most important data cited by various investigators in support of the impurity theory:

1. Two authors (*72, 73*) claimed that purifications of solution media completely eliminates radiocolloid formation. Many others simply stated that purification reduced radiocolloidal aggregation (*47, 48, 58, 59*).

2. Assuming that Bi in tracer quantities can act as an anion or a cation, Werner (*59, 60*) showed that its behavior under the influence of acids, bases, or salts is as would be predicted on the basis of adsorption on impurities. Haissinsky (*5, 69*) criticized this work on the basis of his investigations with macroscopic amounts of Bi and the researches of Paneth and Benjamin on the bismuth anion (*74*), which Haissinsky stated could not exist under Werner's experimental conditions.

3. Coagulation isotherms for various elements under certain conditions show decreases in the per cent coagulated with increases in the concentration of the radioelements (*9, 10, 11, 44*). See section 3 under "The True Colloid Theory" for details.

4. Some radioautographs indicate that impurity centers are present in radiocolloidal aggregates (*6, 70*). See section 6 under "The True Colloid Theory."

Probably the best conception at the present is the one postulated by Chamie and Haissinsky (*44*) combining certain features of both theories. Reference may be made to section 3 under "The True Colloid Theory" for their ideas.

Size Calculations. Three different methods have been used to calculate the sizes of the radiocolloidal aggregates.

1. The first of these methods to be used was that which depends upon diffusion measurements (*5*). In the presence of a large excess of inert electrolyte, it is possible to apply the Einstein-Stokes equation to radiocolloids. This equation takes the form

$$\nu = \frac{RT}{6\pi\eta DN}$$

where D is the diffusion coefficient, R the gas constant, T the absolute temperature, η the viscosity of the medium, N Avogadro's number, and ν the mean radius of the particles.

2. The second method is based upon the quantity of a radiocolloid centrifugable in a given time using a known centrifugal force (*5*). An equation applicable in this situation is

$$\nu = \frac{9\eta \ln (x_2/x_1)}{2\omega^2 t(P_p - P_m)}$$

where t is the time required to bring a particle from position x_1 to position x_2, the x's being distances from the particle to the axis of rotation, P_p and P_m the densities of the particle and the medium, and ω the angular velocity. The symbols η and ν have their previously assigned meanings.

3. Chamie's photographic method (*32, 33*) provides a third method for determining the sizes of radiocolloids. This procedure is especially suited to radioelements emitting particles which leave well-defined tracks in a photographic emulsion. Using a knowledge of the range of particles, direct measurements of the aggregate size may be made under a microscope. The number of atoms in an aggregate may also be approximated. The paths, if not too dense, can be counted, and knowing the

half-life of the nuclide involved plus the exposure time, the number of atoms n in the aggregate may be calculated with the following formula:

$$n = \frac{dN/dt\ t_{1/2}}{0.693}$$

where dN/dt is disintegrations per unit time and $t_{1/2}$ is the half-life. This method has one major difficulty, that being the estimation of the correct value of dN/dt from the number of paths since all particles emitted from an aggregate do not leave observable tracks, particularly those emitted perpendicular to the plane of the emulsion.

APPLICATIONS AND IMPLICATIONS

Radiocolloidal formation may be both helpful and detrimental to the investigator dealing with tracer quantities of radioactive materials. In the following sections some remarks bearing upon these aspects will be presented.

Apparently, two types of adsorption must be dealt with in reference to radiocolloids. The radiocolloidal aggregates themselves will be adsorbed on various surfaces, depending probably upon the charge that they carry. This charge can be regulated by varying the composition of the medium as was done by Godlewski (*37*, *38*, *39*). Thus adsorption of the aggregates may be controlled to some extent. In addition, ions in solution may be adsorbed upon impurities, walls of the container, filter paper, glass, metal plates, or other surfaces with which the solution is brought into contact. Both types of adsorption can be minimized by the addition of a strong acid or a complexing agent.

Nonequivalence. Normally isotopes of identical chemical form behave in almost exactly the same manner under every circumstance, allowing no differentiation of properties between the different isotopes. However, from information already presented demonstrating the difference in behavior of radioactive elements in the forms of ions and of radiocolloids, it would be expected that if a radiocolloid were added to a solution containing a macroscopic amount of an isotope of the same element in the ionic form, the behavior of the two would not be immediately equivalent. Schubert and Conn (*8*) cite an example in which radiocolloidal Zr was added to a strongly acid solution of of inactive Zr. The two forms became equivalent only after a series of chemical reactions. Thus, in order to reach equivalence when a radiocolloid is mixed with a relatively large amount of inactive isotope, it is suggested that several physical and chemical reactions be carried out with the mixture.

Diffusion. Many biological experiments have demonstrated that the reactions and/or uptake of an element in an organism may be a function of the diffusibility of the element. When utilizing carrier-free radionuclides, one should always consider the possibility of the formation of radiocolloids which would alter the diffusibility. Particular attention should be paid to the trivalent and tetravalent elements, since they seem to be very prone to form radiocolloids. Also one should remember that if the conditions exist for the formation of an insoluble compound of the radioactive material being used, then radiocolloidal formation is probable.

Validity of Solubility Products. The discovery of radiocolloids brought serious doubt as to the validity of the solubility product constants of several substances. The solubility principle assumes that a definite state of equilibrium between the solid and liquid phases has been established. Haissinsky (*75*) points out that this state of equilibrium is not attained in the case of radiocolloidal formation. A solution may sit for a period of time without radiocolloidal formation and then through the operation of some unknown factor, the formation process is suddenly initiated. This type of development disturbs the conditions of equilibrium which under ordinary conditions would be established quite rapidly. Also this disturbance of equilibrium is quite slow and therefore it is difficult to decide when a new equilibrium has been reached.

In support of his challenge on the validity of solubility products of a number of substances, Haissinsky (*75*) cites a long list of measurements of solubility products. These data seem to indicate that a number of factors, including age, method of preparation, purity of solvent, method of measurement, and others affect the value of the solubility product obtained with a given substance. It appears that the more insoluble the substance, the more error is possible. Haissinsky maintains that colloid formation below the actual solubility might lead one to believe that true solubility had been attained. Kurbatov and Kurbatov (*9*, *10*) also report deviations from solubility product principles in their investigations. They noted that the per cent Y separated from solution actually increased in going below the recorded solubility product, contrary to what would be expected.

Conditions and Time of Storage. The conditions and time of storage of low concentration radioelements which are liable to form radiocolloids have been shown to be quite important. Chamie and Haissinsky (*44*) reported that Po solutions after aging contained an increased amount of radiocolloid. King and Romer (*62*) showed that storage of Po solutions in paraffin-lined containers prevented radiocolloid formation for long periods of time. Schubert and Richter (*28*) noted that carrier-free Zr-95 stored in 10 N HCl was ionic, while that stored in 1 N HCl was radiocolloidal.

Radiocolloids in Geochemistry. Yagoda (*6*, *70*) has recently reported that radioautographs of quartz or other materials associated with U minerals showed the presence of aggregates of Ra. These aggregates are attributed to radiocolloids. In sampling rocks, these radiocolloidal deposits have led to some difficulty because they are not distributed evenly from sample to sample. The accuracy of age determinations using such minerals is thus placed in doubt.

Yagoda (*70*) cites measurements by Rona in which the Ra content of sea water was found to be 2 to 15

times less than that calculated from the U content. This could hardly be accounted for by concentration processes of algae or plankton, and it was postulated that the Ra was settling out as a radiocolloid or adsorbing on available surfaces.

Isolation of Carrier-Free Nuclides. One of the most useful applications of radiocolloidal formation has been in the separation of carrier-free nuclides from bombarded materials. Without doubt, more separations based on the aggregation of low concentration radioelements will be brought about.

RADIOAEROCOLLOIDS

In her early experiments, Chamie (*76*) observed that aggregates of the active deposit of Rn seemed to form in both dry and moist air. Harrington and Gratias (*30, 77*) sealed Rn into glass tubes with air or other gases and centrifuged the mixtures, after which they showed that the active deposit had precipitated. They went on to find that the formation of groups was enhanced in the presence of polar molecules.

Jedrzejowski (*72*) reported that impurities acted as centers of agglomeration for the radioactive atoms. Harrington (*30, 78*) confirmed the idea that radioaerocolloid formation is facilitated by impurities, but reported that they were not essential. Chamie and Goldstein (*79, 80*) stated that filtration of the Rn does not eliminate the groups.

Harrington and Braaten (*81*) made measurements on the aggregates using the ultramicroscope and photographic plates, coming to the conclusion that they vary widely in their mobilities. The clusters in the active deposits of Rn have been seen with the electron microscope by Martin, Taylor, and Clark, (*82*).

Harrington said that the recoil atoms and ions resulting from radioactive disintegration and the effects of the emitted radiations were prone to remain together upon collision. Polar molecules then adsorb on the group. The aggregate would then continue to grow by further acquisitions. Chamie (*80*) extrapolated these considerations to solutions and said that they could explain the origin and properties of radiocolloids. These ideas imply that polar molecules are necessary for radiocolloid or radioaerosol formation and that radioactivity may be involved in the processes which initiate aggregate formation.

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MOTION PICTURE DEMONSTRATIONS IN ANALYTICAL CHEMISTRY[1]

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CHEMISTRY teachers must not only be good chemists, but specialists in communications as well. Verbal communication indeed has its shortcomings, and these are most noticeable in presenting laboratory techniques. Words are inept for describing operations which require timing and coordination. Here a demonstration by a skilled technician is an unexcelled medium of communication.

The need for demonstrations in analytical laboratories is particularly great, but this order is difficult to fill. A good demonstration requires time for preparation and skill in execution. Furthermore, "live" demonstrations of techniques can be effectively given to only a handful of students at a time. To find personnel for an adequate demonstration program is difficult where large classes are involved.

Motion pictures appear to be our best medium for demonstrations to large audiences. With proper photography, finest details can be shown to the largest classes. Also, a motion picture can be made "foolproof." Mistakes and false moves can be cut out ahead of time. The movies are ready to go at a moment's notice, thus practically eliminating the necessity for time-consuming preparations.

A movie standardizes techniques. It decreases the possibility of teaching inefficient or erroneous techniques. It also leads to uniformity within a class. However, because it ignores many other techniques and implies that the technique shown is the only accepted one, it may be misleading. Among professional chemists, a variety of techniques may be found for any operation. Further, any basic technique is subject to modification upon occasion, depending upon the experiment. Standardization should not be so rigid that modification cannot be readily and gracefully made when necessary. Because a film can efficiently show only a few basic operations, it devolves upon the instructor to expand his student's knowledge and judgment of techniques.

The art and science of producing analytical technique films is three-sided: photographic, communicative, and chemical.

The photographer has special problems. He must be careful of background and must provide adequate lighting. Close-ups, such as those of a balance pointer or an asbestos mat, require special attention. Whether or not the audience will really see the details as intended depends upon the care and judgment of the photographer.

There are a number of tricks to visual communication that enable audiences to get the most from the medium of picture film. For example, an over-the-shoulder shot shows action, as one who was actually carrying out the operation would see it. When viewing such a shot, a student in his mind, projects himself into the action; he carries out the operation.

The photographic and communicative aspects of film making are sufficiently complex to warrant the services of specialists in these fields, if possible. The task then remaining for the chemist is the critical choice of techniques to be shown and proper execution

[1] Presented at the Conference on the Teaching of General and Analytical Chemistry at Oklahoma A. and M. College, June 13, 1952.

of them. Also, the chemist contributes via the sound track explanatory notes as the story unfolds. Much of the verbal explanation is best given by the instructor in the classroom or laboratory situation. The film narrator may conveniently open the way for these comments by referring the audience to the instructor for details at various points in the film.

Demonstration films can contribute to analytical instruction with subjects other than classical laboratory techniques. There are a number of subjects which are increasing in importance and making strong bids to be included in already crowded course curricula. Notable among these is instrumental analysis. Films portraying the principles and applications of modern instruments promise to be an efficient and economical way to introduce the subject in elementary courses. Microorganic combustion analysis could also be demonstrated by film. Procedures which are long and tedious could be shown with the aid of time-collapse technique.

Some beginnings have been made to produce films for demonstrating analytical laboratory techniques. The new tool must yet be tested in the fire of experience. From the tests should come not only improvements, but expansion into a motion picture program adequate for modern analytical curricula.

Acknowledgment

The author wishes to acknowledge the very considerable contribution to this paper by many discussions with Mr. Lewis V. Peterson, Division of Visual Communications, University of Illinois, Urbana, Illinois.

List of Analytical Demonstration Films and Source of Availability

(1) Use of Analytical Balance, Audio-Visual Aids Service, Westbrook Hall, University of Minnesota, Minneapolis 14, Minnesota.

(2) The Analytical Balance, Lewis V. Peterson, Gregory Hall, University of Illinois, Urbana, Illinois.

(3) Techniques of Gravimetric Analysis, Lewis V. Peterson, Gregory Hall, University of Illinois, Urbana, Illinois.

(4) Formation and Handling of Precipitates, M. G. Mellon, Chemistry Department, Purdue University, Lafayette, Indiana. (Not available for distribution.)

A rather complete listing of films for chemical education is available from the American Chemical Society.

SOME (ORGANIC) LAB TIPS

(1) Cracked oyster shells make excellent boiling chips.

(2) A variation of the "Cold Finger" may be constructed by placing a 6- × 3/4-inch test tube with side arm, in a 125 cc. Erlenmeyer flask. The sizes may be varied if desired, the tube is held secure by three or four rubber washers fitted around the circumference of the tube. This setup minimizes the danger of blow outs.

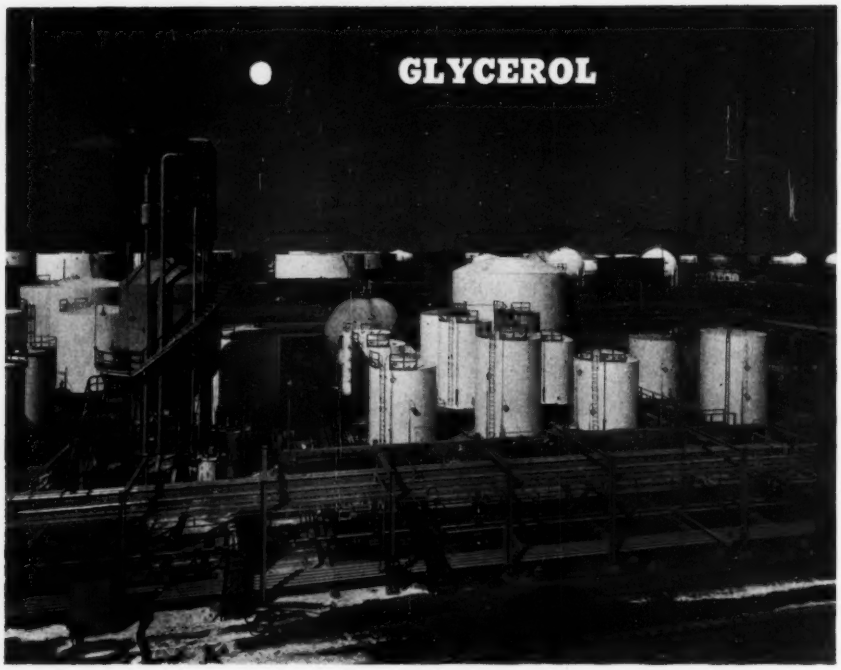
(3) One can increase the capacity of a stopper or cork by simply inserting a thistle tube in one hole. At the top of the thistle, another cork or stopper is inserted containing two or three holes. For example, when using a distilling flask in which a two hole stopper is placed, a stirring rod may be attached through one hole, and a thermometer and separatory funnel placed in the stopper atop the thistle tube which is inserted in the other hole.

(4) Midget motors which sell for two or three dollars (sold by merchandise houses) are excellent substitutes for more expensive stirrers. They do not operate on standard current but rather run on flashlight cells.

(5) Rubber plugs (the type used in bathtubs) from which the rings have been removed can be used in place of rubber stoppers or corks especially for flasks 500 cc. and over. They are easy to bore, and 4, 5, or even 6 holes may be bored in them with little effort.

—Marvin Antelman. Yeshiva University, New York, New York.

GLYCEROL

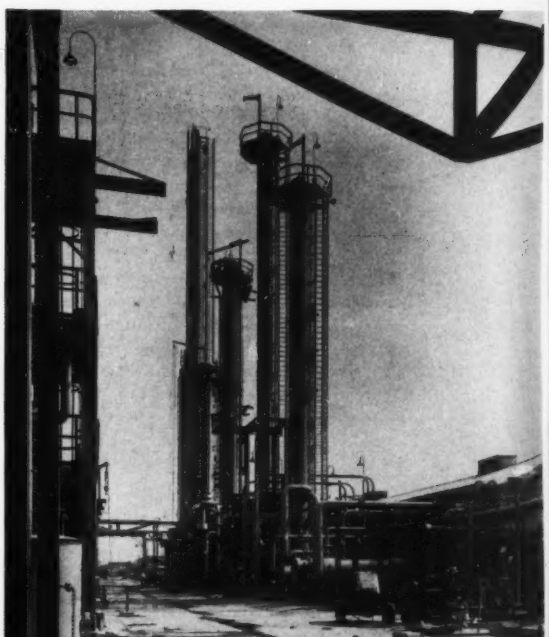


Courtesy of Shell Chemical Corporation

Synthetic Glycerol Plant, Houston, Texas

ELBERT C. WEAVER

Phillips Academy, Andover, Massachusetts



Courtesy of Shell Chemical Corporation

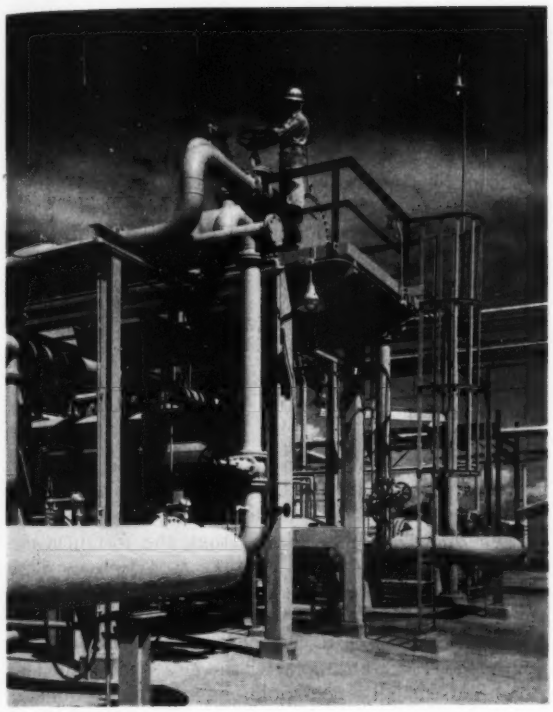
Fractionating Columns

IF YOUR students in elementary organic chemistry should represent the addition of chlorine to propylene as

$$CH_3{>}CH{\cdot}CH_3 + Cl_2 \rightarrow CH_2{=}CH{\cdot}CH_2Cl + HCl$$

showing the formation of allyl chloride, you might mark this atypical equation as incorrect. Nevertheless, the reaction is the first step in a successful commercial synthesis of glycerol. In this process, flow rate, temperature, and pressure are carefully controlled. Many years of investigation were needed to bring this reaction into the rage of commercial practicability. Now a plant is in operation that produces glycerol from hydrocarbons.

The second and third steps of this process, carried out simultaneously, include complete chlorination of allyl chloride to trichloropropane and the replacement of halogen by the hydroxyl group, which in turn is derived from sodium hydroxide solution in water solution.



Portion of Allyl Chloride Synthesis Unit. Here Allyl Chloride is Made from Propylene and Chlorine.

The conversion takes place under controlled conditions of pH, flow rate, temperature, and time. The dilute solution of glycerol and common salt is evaporated and desalted. The glycerol is recovered by distillation under reduced pressure. After other steps of purification, the glycerol comes to the market 99 per cent pure.

Over 1500 uses of glycerol are known in the arts, but today three major uses account for the principal applications. Explosives, cellophane, and alkyd resins are the major outlets today. Even ten years ago, the manufacture of alkyd resins was the principal use of glycerol. Over 40 million pounds were consumed for the purpose. Glycerol imparts flexibility and toughness to cellophane, which otherwise would be a brittle and unsatisfactory wrapping material. The use of glycerol in the manufacture of explosives is well known. The total demand is in excess of 200 million pounds for all uses, a demand that could not be met from the familiar source, the by-product of saponification of fats, alone.

$$CH_2{=}CH{\cdot}CH_2Cl + Cl_2 \rightarrow CH_2Cl{\cdot}CHCl{\cdot}CH_2Cl$$

$$CH_2Cl{\cdot}CHCl{\cdot}CH_2Cl + 3NaOH \rightarrow C_3H_5(OH)_3 + 3NaCl$$

• THERMITE IGNITION ASSURED

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University of Toronto, Toronto, Canada

THE COMFORTABLE, safe, and surefire time fuse for the thermite reaction described in this journal by O. C. Klein[1] leaves nothing to be desired by anyone who places a premium on reliability in demonstrating it to lecture or laboratory groups. On the numerous occasions I have used this means to initiate the reaction, it has never failed to respond exactly as intended.

This ingenious arrangement, however, is not always able, even with the igniter mentioned, to ignite some commercial grades of thermite which are so coarse as to be merely puffed away from the site of the flash without being set off. In order to guarantee the ignition of such thermite, it is well to emphasize that a good intermediate igniter is essential.

For this purpose an intimate mixture of equal volumes of finely powdered aluminum and red oxide of iron, Fe_2O_3, both readily available from the stockroom shelf, has been found excellent, no failure having been experienced with it in long use.

It is suggested, therefore, that anyone who desires a trouble-free demonstration of the thermite reaction prepare it as follows: into a crater in the thermite pour a few spoonfuls of this intermediate igniter mixture, crowding it about somewhat with the surrounding thermite; and into a smaller crater in this intermediate igniter pour the BaO_2–aluminum powder mixture, followed up with the potassium permanganate and the drop of glycerine exactly as previously recommended[1]. The whole chain reaction, once started, is explosively rapid.

The demonstration now becomes one to be shown with confidence, not with misgivings about misfire. Be sure to stand well away, once the glycerine is added; and it may be just as well to have the front row in the lecture room vacated, lest someone be burned by the flying globules of molten iron.

If the BaO_2–aluminum powder igniter is not purchasable, thoroughly stir together approximately equal volumes of the two powdered ingredients, guarding against chance sparks or flame getting at it. For the sake of added safety, it is better to use a very long stirring rod and to prepare only a few grams of the mixture at a time, as needed.

[1] KLEIN, O. C., "An Improved Method for Igniting Thermite Reactions," J. CHEM. EDUC., **14**, 320 (1937).

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

pH APPROXIMATIONS

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THE pH of solutions may be approximated by calculations. The data necessary for many of these calculations are given in the tables included in this paper. Such calculations are not to be used in place of actual pH determinations, but they may lead to an understanding of some of the problems of pH, and may sometimes help in the solution of these problems. In general, it may be stated that the formulas presented in this paper do not apply to extremely dilute or extremely concentrated solutions. In the former the dissociation of water is a major factor, and as the concentration becomes less than 10^{-7} molar, the pH approaches 7.0. In the latter the activities of the ions, which are usually negligible, have a great influence on the pH. The approximations are very good for pure chemicals if the molar concentration is not greater than one molar or less than 10^{-6} molar or if the pH calculation lies between 2.0 and 12.0. Many of the compounds on the market contain free acid or free base, with the result that they may have a more desirable pH or that they may be more stable. Since the theoretical pH is for the pure chemical, preparations that contain free acid or free base will have a pH different from that obtained by calculation based on a pure substance. Sodium salicylate, a salt of a weak acid and a strong base, should theoretically have a pH greater than 7.0. The United States Pharmacopeia states that this salt is neutral or slightly acid to litmus; and the pH is given as 5.0 to 6.0. The calculated pH of a 0.1 molar solution of sodium salicylate is 8.0. The measured pH of a 0.1 molar solution of a sample labeled *Sodium Salicylate U. S. P.* was 6.7.

TABLE A
Dissociation Values of Acids

	A	A'		A	A'
Acetic	2.4	...	Hypophosphorous	1.0	...
Acetylsalicylic	1.7	...	Iodic	C	...
Alanine	4.9	...	Lactic	1.9	...
p-Aminobenzoic	2.5	...	Maleic	1.0	3.1
Arsenic	1.2	3.5	Malic	1.7	2.5
	(A'' = 4.6)		Malonic	1.4	3.1
Arsenious	4.6	...	Mandelic	1.7	...
Barbituric	2.0	...	Naphthoic	2.0	...
Benzoic	2.1	...	Nicotinic	2.4	...
Boric	4.6	...	Nitrous	1.7	...
Cacodylic	3.2	...	Oxalic	C	2.1
Camphoric	2.3	2.6	Perchloric	C	...
Caproic	2.4	...	Periodic	C	...
Caprylic	2.5	...	Permanganic	C	...
Carbonic	3.2	5.1	Phenol	5.0	...
Chloric	C	...	Phenylacetic	2.2	...
Cinnamic	2.0	...	Phosphoric	1.1	3.6
Citric	1.5	2.4	.....	(A'' = 6.0)	
	(A'' = 2.7)		Phosphorous	C	3.1
Diethylbarbituric	3.7	...	Phenolphthalein	4.9	...
Diphenylacetic	2.0	...	Phthalic	1.5	2.7
Formic	1.9	...	Picric	C	...
Fumaric	1.5	2.2	Propionic	2.4	...
Gallic	2.2	...	Saccharin	C	...
Glutamic	4.9	...	Salicylic	1.5	6.7
Glutaric	2.2	...	Stannic	4.7	...
Glycerophosphoric	C	3.1	Succinic	2.1	2.8
Glycine	4.9	...	Sulfanilic	1.6	...
Glycolic	1.9	...	Sulfuric	C	1.0
Hippuric	1.8	...	Sulfurous	C	3.6
Histidine	4.6	...	Tartaric	1.5	2.3
Hydriodic	C	...	Thioglycolic	1.8	...
Hydrobromic	C	...	Thiosulfuric	1.0	...
Hydrochloric	C	...	Toluic	2.0	...
Hydrocyanic	4.6	...	Trichloroacetic	C	...
Hydrofluoric	1.6	...	Uric	2.0	...
Hydrosulfuric	3.6	7.5	Valeric	2.4	...
Hydroquinone	5.0	...	Vanillic	2.3	...
Hypochlorous	3.7	...			

A = ½ pK_a for 1st stage; A' for 2nd stage and A'' for 3rd stage.

DEFINITIONS

As an aid in the understanding of the formulas of this paper the following definitions and symbols are presented at the beginning.

A = ½pK_a = $-$½ log K_a, where K_a is the dissociation constant for the first stage of ionization of the acid.
K_a' and K_a'' indicate, respectively, the second and the third stages of ionization of the acid.
A' and A'' designate, respectively, the A value of K_a' and K_a''.
B = ½pK_b = $-$½ log K_b, where K_b is the ionization constant of the base.
C = ½pc = $-$½ log c, where c is the molar concentration.
c_a, c_b, and c_s designate, respectively, the molar concentration of the acid, base, and salt.
C_a, C_b, and C_s designate, respectively, the C value of the acid, base, and salt.

TABLE B
Dissociation Values of Bases

	B		*B*
Acetanilid	6.7	Leucine	5.8
Aconitine	2.9	Lithium hydroxide	*C*
Alanine	5.6	Magnesium hydroxide	*C*
Aminopyrine	4.6	Morphine	3.1
Ammonium hydroxide	2.4	Narceine	5.4
Aniline	4.7	Narcotine	3.9
Apomorphine	3.5	Nicotine	3.1
Arsenous oxide	2.0	Papaverine	4.1
Atropine	2.2	Physostigmine	3.1
Barium hydroxide	*C*	Pilocarpine	3.6
Caffeine	1.7	Piperidine	1.4
Cinchonidine	2.9	Piperine	7.0
Cinchonine	2.9	Procaine	2.6
Calcium hydroxide	*C*	Pyridine	4.4
Cocaine	2.8	Potassium hydroxide	*C*
Codeine	3.0	Quinidine	2.7
Colchicine	6.2	Quinine	3.0
Coniine	1.5	Quinoline	4.6
Emetine	2.9	Silver hydroxide	2.0
Ephedrine	2.3	Sodium hydroxide	*C*
Eserine	3.1	Sparteine	1.1
Ethanolamine	2.3	Strontium hydroxide	*C*
Ethylenediamine	2.0	Strychnine	3.0
Ethylmorphine	3.1	Theobromine	6.7
Glutamic acid	5.8	Thiazole	5.7
Glycine	5.8	Thiourea	7.5
Histidine	4.0	Urea	6.9
Hydrastine	3.9	Veratrine	2.6
Hydroquinone	2.7	Xanthine	6.7
Lead hydroxide	1.5	Zinc hydroxide	2.2

$B = {}^1/_2\ pK_b$ for the base.

$pK_w = -\log K_w$, where K_w is the ionic product of water.
$pH = 14 - pOH = -\log c_{H^+}$, where c_{H^+} is the molar concentration of the hydrogen ions.
$pOH = -\log c_{OH^-}$, where c_{OH^-} is the molar concentration of the hydroxyl ions.
pI = Isoelectric point.

Weak acids and weak bases do not dissociate completely. In order to approximate the pH of these the dissociation constant must be known and must be considered in the calculation. Tables A and B list respectively the *A* and *B* values of some of the important acids and bases. These values may be substituted directly into the formulas. The dissociation values of chemicals not listed in these tables may be obtained from Handbooks and "International Critical Tables" (*1*). If the values in the literature are given in the form $-\log K$ or p*K*, they may be converted to *A* or *B* values by dividing them by two. If the values are given as exponential expressions, Table C is used to convert them to *A* or *B* values. The conversion factors given in Table C may be used to convert exponential expressions to the form required in the formulas. The basic formulas used in the development of the simplified formulas presented in this paper may be found in textbooks (*2*, *3*). The simplified formulas are summarized in Table D.

Weak Acids. For a weak acid the hydrogen ion concentration and the pH may be calculated from the following equations:

$$c_{H^+} = (cK_a)^{1/2}$$

Taking negative logs,

$$-\log c_{H^+} = -{}^1/_2 \log c - {}^1/_2 \log K_a$$

Therefore,

$$pH = C + A$$

Example: The pH of 0.1 *M* boric acid is calculated:

$$pH = C + A = 0.5 + 4.6 = 5.1$$

Values: U. S. P., 5.1 Measured, 5.4

Weak Bases. Similarly with a weak base it can be shown:

$$c_{OH^-} = (cK_b)^{1/2}$$

$$-\log c_{OH^-} = -{}^1/_2 \log c - {}^1/_2 \log K_b$$

$$pOH = C + B$$

$$pH = 14 - B - C$$

Example: The pH of 0.1 *M* ammonia water is calculated:

$$pH = 14 - B - C = 14 - 2.4 - 0.5 = 11.1$$

Values: U. S. P., 11.3 Measured, 11.1

Strong Acids. To approximate the pH of solutions of strong acids or of strong bases, the dissociation may be considered to be complete. The hydrogen ion concentration of a strong acid is considered to be equal to the molar concentration of that acid. The pH of the acid is equal to the negative logarithm of the hydrogen ion concentration.

$$pH = -\log c_{H^+}$$

For 0.1 *M* hydrochloric acid $c_{H^+} = 1 \times 10^{-1}$

$$pH = -\log (1 \times 10^{-1}) = -(-1) = 1.0$$

This calculation may be accomplished in another manner. Since, $-{}^1/_2 \log c = C$, then $-\log c = 2C$.

The formula for the pH of a weak acid given previously could also be used.

$$pH = C + A$$

TABLE C
Conversion Factors

	0.8–1.2	1.3–1.9	2.0–3.1	3.2–4.9	5.0–7.9	8.0–12
10^0	0.0	−0.1	−0.2	−0.3	−0.4	−0.5
10^{-1}	0.5	0.4	0.3	0.2	0.1	0.0
10^{-2}	1.0	0.9	0.8	0.7	0.6	0.5
10^{-3}	1.5	1.4	1.3	1.2	1.1	1.0
10^{-4}	2.0	1.9	1.8	1.7	1.6	1.5
10^{-5}	2.5	2.4	2.3	2.2	2.1	2.0
10^{-6}	3.0	2.9	2.8	2.7	2.6	2.5
10^{-7}	3.5	3.4	3.3	3.2	3.1	3.0
10^{-8}	4.0	3.9	3.8	3.7	3.6	3.5
10^{-9}	4.5	4.4	4.3	4.2	4.1	4.0
10^{-10}	5.0	4.9	4.8	4.7	4.6	4.5
10^{-11}	5.5	5.4	5.3	5.2	5.1	5.0
10^{-12}	6.0	5.9	5.8	5.7	5.6	5.5
10^{-13}	6.5	6.4	6.3	6.2	6.1	6.0
10^{-14}	7.0	6.9	6.8	6.7	6.6	6.5

This table may be used to convert *c* to *C*, K_a to *A*, and K_b to *B*. Example: $0.0024 = 2.4 \times 10^{-3}$. Read across the row to 2.0–3.1, then down this column to the row designated 10^{-3}. The answer is 1.3.

If with a strong acid the A in this formula equals C, then the formula for a weak acid may be used. The A values of strong acids in Table A are given as equal to C. For the problem above refer to Table A; the value for hydrochloric acid is given as C. By referring to Table C, 1×10^{-1} is seen to have a numerical value of 0.5. By substitution then,

$$\text{pH} = C + A = C + C \text{ or } 2C = 2(0.5) = 1.0$$

Values: U. S. P., 1.0 Measured, 1.2

The calculation for the hydrochloric acid problem above is simple enough to be easily handled without resorting to the formula or the tables.

Assume that the acid is 0.16 M then the c_{H^+} is 1.6×10^{-1}, $\text{pH} = -\log(1.6) + -\log(10^{-1}) = -0.2 + 1.0 = 0.8$. From Table A, A equals C, and from Table C, 1.6×10^{-1} equals 0.4. Then

$$\text{pH} = 2C = 2(0.4) = 0.8$$

Value: Measured, 1.0

Strong Bases. For strong bases it follows that

$$\text{pOH} = -\log c_{OH^-} = 2C \text{ thus } \text{pH} = 14 - 2C$$

The formula for weak bases may be used instead of the above formulas by making B in Table B equal to C.

Example:

The pH of Milk of Magnesia U. S. P. is calculated: For pH approximation this suspension may be considered a saturated solution of magnesium hydroxide. The solubility of magnesium hydroxide is 1.5×10^{-4} moles per liter. From Table B, B equals C and 1.5×10^{-4} is found to be equal to 1.9 from Table C

$$\text{pH} = 14 - B - C = 14 - C - C = 14 - 1.9 - 1.9 = 10.2$$

Values: U. S. P., 10.6 Measured, 10.1

Salts. The approximation of the pH of the end point in a neutralization titration is one of the important applications of these formulas. The pH of a salt that is formed in a neutralization is the same as the pH value of the end point of the titration.

Salts of strong acids and strong bases do not undergo hydrolysis. The pH of solutions of such salts may thus be considered to be 7.0. The solutions may be referred to as "neutral." Most other salts, when dissolved, are ionized and dissociated to some degree. The pH produced as a result of hydrolysis may be approximated. The method of making the approximate calculation is given in a later section.

Acid Salts. For an acid salt of the type MHA and MH_2A

$$\text{pH} = -\log(K_a K_a')^{1/2}$$

$$\text{pH} = -{}^1/_2 \log K_a - {}^1/_2 \log K_a'$$

$$\text{pH} = A + A'$$

Example: The pH of 0.1 M sodium bicarbonate is calculated:

$$\text{pH} = A + A' = 3.2 + 5.1 = 8.3$$

Values: U. S. P., 8.2 Measured, 8.2

Similarly an acid salt of the type M_2HA

$$\text{pH} = -\log(K_a' K_a'')^{1/2}$$

$$\text{pH} = A' + A''$$

Example: The pH of 0.1 M disodium hydrogen phosphate is calculated:

$$\text{pH} = A' + A'' = 3.6 + 6.0 = 9.6$$

Values: U. S. P., 9.2 Measured, 9.1

Salt of a Strong Base and a Weak Acid. The salts of strong bases and weak acids hydrolyze to give a basic solution. The reaction of the anion of a weak acid with water to form the undissociated acid and hydroxyl ion makes this solution basic.

$$\text{pH} = {}^1/_2 \text{p}K_w + {}^1/_2 \text{p}K_a + {}^1/_2 \log c$$

$$\text{pH} = 7.0 + A - C$$

Example: The pH of 0.1 M potassium cyanide is calculated:

$$\text{pH} = 7.0 + A - C = 7.0 + 4.6 - 0.5 = 11.1$$

Value: Measured, 10.8

Salt of a Weak Base and a Strong Acid. The salts of weak bases and strong acids hydrolyze to give an acidic solution. The reaction of the cation of the weak base with water to form the undissociated base and the hydronium (hydrogen) ion makes this solution acidic.

$$\text{pH} = {}^1/_2 \text{p}K_w - {}^1/_2 \text{p}K_b - {}^1/_2 \log c$$

$$\text{pH} = 7.0 - B + C$$

Example: The pH of 0.1 M ammonium chloride is calculated:

$$\text{pH} = 7.0 - B + C = 7.0 - 2.4 + 0.5 = 5.1$$

Values: U. S. P., 4.6 Measured, 5.4

Salt of a Weak Base and a Weak Acid. A solution of a salt of a weak acid and a weak base will be considerably hydrolyzed; and if the acid is stronger than the base, the solution will be acidic. Conversely, if the base is stronger than the acid, the solution will be basic. Amino acids and other ampholytes may be considered to be salts of weak acids and weak bases. For amino acids, the pH of the pure amino acid is also the isoelectric point, the pH at which the basic and acidic ionizations of an ampholyte take place to the same extent.

$$\text{pH} = {}^1/_2 \text{p}K_w + {}^1/_2 \text{p}K_a - {}^1/_2 \text{p}K_b = \text{pI}$$

$$\text{pH} = 7.0 + A - B = \text{pI}$$

Example: The pH of 0.1 M ammonium acetate is calculated:

$$\text{pH} = 7.0 + A - B = 7.0 + 2.4 - 2.4 = 7.0$$

Value: Measured, 6.8

Example: The pH of 0.1 M glycine is calculated:

$$\text{pH} = 7.0 + A - B = 7.0 + 4.9 - 5.8 = 6.1$$

Values: Handbook, 6.1 Measured, 6.5

Buffers. A buffer solution is one which resists a change of pH upon the addition of acid or alkali. Buffer solutions consist of a mixture of weak acid and its salt, or of a weak base and its salt, or of an acid salt, or of a salt of a weak acid and a weak base. Approximations of the pH of the last two types of salts have been discussed previously.

Weak Acid and Its Salt.

$$pH = pK_a + \log(c_s/c_a) = pK_a + \log c_s - \log c_a$$

$$pH = 2A - 2C_s + 2C_a$$

Example: The pH of a solution 0.1 M sodium acetate in 0.05 M acetic acid is calculated:

$$pH = 2A - 2C_s + 2C_a = 4.8 - 1.0 + 1.2 = 5.0$$

Value: Measured, 5.0

Example: The pH of U. S. P. Phosphate Buffer pH 8.0 is calculated:

By calculation a liter of this buffer solution is found to contain 3.2×10^{-4} moles KH_2PO_4. This is considered to be the "acid," $(H_2PO_4)^-$, with a C_a value of 1.7 and an A value of 3.6. A liter of the buffer is calculated also to contain the equivalent of 2.2×10^{-3} moles $NaKHPO_4$. This is considered to be the salt with a C_s value of 1.3.

$$pH = 2A + 2C_a - 2C_s = 7.2 + 3.4 - 2.6 = 8.0$$

Weak Base and Its Salt.

$$pH = -\log(K_w c_s/K_b c_b) = \log(K_b c_b/K_w c_s)$$

$$pH = \log K_b + \log c_b - \log K_w - \log c_s$$

$$pH = -2B - 2C_b + 14 + 2C_s$$

$$pH = 14 - 2B - 2C_b + 2C_s$$

Example: The pH of a solution of 0.1 M ammonium chloride in 0.05 M ammonium hydroxide is calculated:

$$pH = 14 - 2B - 2C_b + 2C_s = 14 - 4.8 - 1.2 + 1.0 = 9.0$$

Value: Measured, 9.1

TABLE D
Formulas for pH Calculations

1.	Acids	$pH = A + C$
2.	Bases	$pH = 14 - B - C$
	Salts	
3.	Acid MHA or MH_2A	$pH = A + A'$
4.	Acid M_2HA	$pH = A' + A''$
5.	Strong base–weak acid	$pH = 7.0 + A - C$
6.	Strong acid–weak base	$pH = 7.0 - B + C$
7.	Weak base–weak acid	$pH = 7.0 + A - B = pI$
	Buffers	
8.	Weak acid and its salt	$pH = 2A - 2C_s + 2C_a$
9.	Weak base and its salt	$pH = 14 - 2B - 2C_b + 2C_s$

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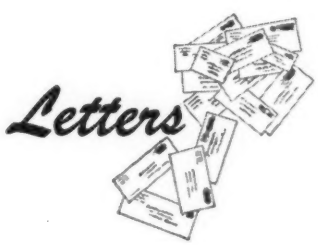
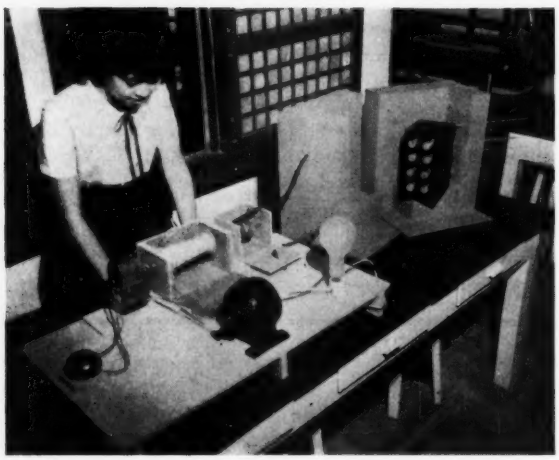


To the Editor:

In the February, 1952, issue of the JOURNAL I read with great interest the article on "Dynamic nuclear reaction models" by Van Dam, Lawrence, and Shapiro of the University of Illinois. I was especially attracted by the illustration of the model of the atomic pile because I realized that our own model here in this institution can favorably compare with it, if I may modestly say so.

I enclose a picture of our model. You will see that it is open for illustration purposes, but the shield is so constructed that it can be completely closed to enclose the pile. All materials are made of wood but painted so as to imitate concrete, graphite, aluminum (covering of uranium rods), etc., respectively. A hidden electrical device sets the motor working and the bulb glowing. A belt connected from the motor to the miniature turbine moves the small generator which causes the electric bell to ring. For the student and onlooker, however, the whole model, which aims at showing the uses of an atomic pile, reads thus: A suitable fluid surrounding the pile in tubes leaves the pile superheated, and transforms the water in the coils which is connected to the turbine, into steam. The steam causes the turbine to rotate, and the turbine then runs the generator which causes the motor to rotate, the bulb to glow, and the bell to ring. Provision is also made (which cannot be



seen in the picture) for a point at which the condensed steam from the coil is returned to the coils, and for the radioactive fluid to be disposed of and replaced.

I am sending you the picture of this model in the hope that it may find a place in your JOURNAL, and that it might be of interest to your readers to hear about instructional activities in chemistry in this remote place. We use the Journal widely in this department, and follow its guidance and suggestions in many ways.

SISTER M. LIGUORI, O.S.B.
ST. SCHOLASTICA'S COLLEGE
MANILA, PHILLIPPINE ISLANDS

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AN INTRODUCTION TO ORGANIC CHEMISTRY

Alexander Lowy **and** ***Benjamin Harrow.*** **Revised by** ***Benjamin Harrow,*** **Professor of Chemistry, and** ***Percy M. Apfelbaum,*** **Associate Professor of Chemistry, College of the City of New York. Seventh edition. John Wiley & Sons, Inc., New York, 1951. xiv + 480 pp. Illustrated. 14.5 × 22 cm. $5.**

THIS most recent edition of a long-known textbook follows generally the plan of its many predecessors. The scope and also much of the wording is essentially unchanged. The time-honored division of aliphatic and aromatic compounds is retained. In addition to the chapters almost universally found in organic textbooks, there are chapters on Nucleoproteins, Pyrimidines, and Purines; Synthetic Polymers, Plastics and Rubbers; Dyes; Terpenes and Related Substances; Alkaloids; Arsenic, Mercury, and Bismuth Compounds of the Aromatic Series; A Brief Review of Common Type Reactions; Plant and Animal Pigments; Enzymes, Vitamins, Hormones; and Nomenclature of Organic Compounds.

The treatment seems to be slanted somewhat in the direction of medicine and the various biological sciences. Also, the emphasis seems clearly to be more upon descriptive chemistry than upon theory. The previous editions have been found useful both in one-semester courses and the more usual two-semester courses. Doubtless the present edition also will find application in some courses of each length, since it is more extensive than the typical books written specifically for one-semester courses, and less extensive than most of the books usually used in the year course. The factual content actually possibly approximates more nearly what may reasonably be covered in the longer course.

The book is very neatly printed, and for the most part is quite readable. The nomenclature is good, and there is a reasonably adequate discussion throughout the book of nomenclature principles. Much of this material is then pulled together in the final chapter, which is devoted exclusively to nomenclature. This chapter, however, consists primarily of a long list of names of radicals and of ring systems. This is fine reference material, but will not in the opinion of the reviewer be particularly useful to beginners.

Those who have found the previous editions adapted to their needs should find the present edition adequate.

LAWRENCE H. AMUNDSEN
UNIVERSITY OF CONNECTICUT
STORRS, CONNECTICUT

EXHAUST HOODS

J. M. DallaValle, **Professor of Chemical Engineering, Georgia Institute of Technology. Second edition. The Industrial Press, New York, 1952. vi + 146 pp. 127 figs. 30 tables. 15.5 × 23.5 cm. $3.50.**

THIS is a book for ventilation and chemical engineers interested in the fundamentals of aerodynamics applied to removal of contaminated air from a variety of type installations. Starting with chapters on Theory of Flow of Gases Into an Opening, Velocity Characteristics. . . ., Velocity Contours. . . ., the author proceeds to consider topics classified by hood types, or general purposes, rather than specific applications. Dusts, mists, fumes, sawdust, flour, etc., serve as central topics.

This revised work goes much beyond the first edition which was mostly a symposium report of limited scope. Many new items have been added from industrial and government sources. Problems are presented with solutions illustrating calculations in hood design.

It is evident that the author has considered laboratory hoods as a special application outside the limits of his work. This is unfortunate in view of the importance of this topic in both academic and industrial circles. The book is thus of little interest to the large number of persons who really are novices in this business—for example, the building committee and the local architect who gets the laboratory design job. It would seem to be highly advisable for the author, in his third edition, to discuss

some of the mooted questions, such as the choice between open versus window-fitted hoods, location and number of fans in multiple laboratory installations, disposal of effluent in populous areas near hoods, and particularly a critical appraisal of relative merits of available lining and flue materials.

Numerous and excellent line drawings as well as half-tones give thorough illustration of design, with contributions from many American industries. Paper quality, printing, proofreading, and binding are satisfactory. The book is unique, and is an obvious "must" to a ventilation engineer, but not to ordinary laboratories of "pure" chemistry.

G. ROSS ROBERTSON

UNIVERSITY OF CALIFORNIA
LOS ANGELES

● ALKALI SOILS

W. P. Kelley, **Professor Emeritus, Soil Chemistry, University of California, Berkeley. Reinhold Publishing Corp., New York, 1951. vi + 176 pp. 14 figs. 40 tables. 16 × 23.5 cm. $5.**

IN SCOPE this monograph primarily treats with the soil itself and the effects of soluble salts brought into the soil. The irrigation and drainage relationships are also discussed at some length.

The author has organized the material in a most logical manner, discussing first the origin and accumulation of soluble salts in nature. This is followed by a description of the effects of soluble salts on the soil and their role in base exchange or cation exchange. The modern concept of the genesis and evolution of alkali soils naturally follows these introductory chapters.

The latter half of the book is largely devoted to the more practical aspects of the subject and considers the relation of salt composition of irrigation water to alkali soils; the effect of soils, or more specifically soluble ions and adsorbed ions, on growth of plants; and the reclamation of alkali soils.

The reviewer believes the author has accomplished his objective in writing this monograph, which will be of value to and is primarily intended for scientists and teachers of soils and agronomy, students of soils, and irrigation, drainage, and soil conservation experts in arid and semiarid regions. Since sufficient background material and definitions are presented to make the subject understandable for those with limited scientific training it should be of value to agricultural county agents and farm advisors.

The quality of binding and paper is excellent. Large, easily readable type has been used and the subject matter is well supplemented with tables, graphs, and a few photographs. A selected bibliography of the more important articles and books in this field (150 in all) has been appended. Proofreading was quite good; only eight typographical errors were noted.

E. A. FIEGER

LOUISIANA STATE UNIVERSITY
BATON ROUGE, LOUISIANA

● THE FISCHER-TROPSCH AND RELATED SYNTHESES

Henry H. Storch, Norma Golumbic, **and** ***Robert B. Anderson,*** **respectively Chief, Technical Assistant, and Physical Chemist, Research and Development Branch, Office of Synthetic Liquid Fuels, U. S. Bureau of Mines. John Wiley & Sons, Inc., New York, 1951. xii + 610 pp. Illustrated. 15 × 23 cm. $9.**

THE authors state that this book is the result of an extensive and critical review of the large number of reports and documents pertaining to the selective catalytic hydrogenation of carbon monoxide, or the Fischer-Tropsch and related syntheses, for the production of aliphatic hydrocarbons and alcohols. Although many of the documents are reports of inspection of German plants and interrogation of German scientists, the book has made complete use of the American literature through 1950.

The book presents a fundamental approach to this particular industrial synthesis, an approach built on the thermodynamics of the reaction, its kinetics, and the mechanism of catalytic activity. Certain chapters or sections of the book are excellent general reviews of these phases of physical chemistry.

After a brief introduction on the structure and reactivity of the reactants, carbon monoxide, and hydrogen, an introduction (77 pages) to heterogeneous catalysis prepares the reader for a specific discussion (207 pages) of Fischer-Tropsch catalysts. This is naturally followed by the process development (118 pages) discussing reactor design, heat transfer, and other engineering problems. A very short (22 pages) discussion is given of the oxo-process and isoparaffin synthesis (more material here certainly would be welcomed). The concluding chapter (130 pages) is devoted to the kinetics and reaction mechanism of the reactions.

The authors have performed a mighty task in bringing together all the captured information and combining this skillfully with the facts of the known literature and their own work in the U. S. Bureau of Mines. The application of fundamental knowledge to an industrial problem is stressed in this book.

KENNETH A. KOBE

UNIVERSITY OF TEXAS
AUSTIN, TEXAS

● ABSORPTION AND EXTRACTION

Thomas K. Sherwood, **Dean of Engineering, Massachusetts Institute of Technology; and** ***Robert L. Pigford,*** **Chairman, Department of Chemical Engineering, University of Delaware. Second edition. McGraw-Hill Book Co., Inc., New York, 1952. ix + 478 pp. 213 figs. 32 tables. 15 × 23.5 cm. $7.50.**

THIS new edition of a basic text is evidently the product of a dilemma. As noted in the preface, the field of diffusional operations is rapidly approaching the point where the basic transport operations of heat, mass, and momentum transfer may be developed from a single unified theory of turbulence mechanism. However, the present state of knowledge is still somewhat shy of this desired goal, and it has been necessary for the authors to compress a large body of new knowledge of theoretical and applied diffusion into the framework of the 1937 edition. The bulges are quite noticeable.

Although the title of the text implies limitation to only two unit operations the actual coverage of the material is much broader. Certainly the fundamental theories of transfer developed in the first four chapters may be applied to analysis of any process involving interphase transfer. Chapter IV summarizes—parenthetically—the basic theory for processes involving simultaneous heat and mass transfer such as humidification, dehumidification, and drying. While pertinent to the rigorous treatment of adiabatic absorption columns, this chapter is an integrated and self-contained exposition of its subject matter and gives needed emphasis to the new "enthalpy potential" methods. Much of the material on absorber design is equally applicable to distillation column design, and Chapter IX on simultaneous absorption with chemical reaction should be of interest to physical chemists.

Unfortunately, the organization and presentation of the material in several chapters is spotty and obscure. Many of the derivations have been overcondensed, and the nomenclature is occasionally arbitrary and confusing. The number of typographical and numerical errors is inordinately high for a basic text, and too many of the frequent illustrative examples fail to achieve their purpose. The necessary forced wedding of empiricism to theory in the chapters on design leads to an uneven, hybrid development of these sections. For service as a class text on the graduate level, this book should be accompanied by a set of explanatory notes.

The total number of references included in the extensive bib-

liography has been increased from the 200 of the first edition to 518 in the present edition. The number of pages has similarly grown from 278 to 475, and this is indicative of the extent of increased coverage afforded by the new edition. Expansion of the sections on basic theory, injection of the economic approach to absorber design, and thorough revision of the original material in the first edition has resulted in the production of a new book, rather than a new edition. The book is exhaustively thorough, comprehensive, and—to date of publication—complete. "Absorption and Extraction" will serve as an excellent source book for both researchers and engineers in the many fields of interphase diffusional operations.

BERNARD J. LERNER

THE UNIVERSITY OF TEXAS
AUSTIN, TEXAS

● VALENCE

C. A. Coulson, Rouse Ball Professor of Mathematics in the University of Oxford. Oxford University Press, New York, 1952. vii + 338 pp. Illustrated. 21.5 × 14 cm. $5.

IN THE opinion of this reviewer, no one has done more to popularize the molecular orbital theory of valence and its application to organic chemistry than C. A. Coulson through his review articles in the *Proceedings of the Royal Society of Edinburgh*, in *Endeavour*, and in the *Quarterly Reviews of the Chemical Society*. Hence it was expected that his new book, "Valence," would be written in such a way that most chemists could understand it without difficulty. This hope was fostered by the statements in the Preface that students of chemistry "should be sufficiently acquainted with the chief ideas and the essential tools that lie behind the modern theory of valence" and that "practically no mathematics is needed for this purpose, since almost everything necessary can be put in pictorial terms." This promise is not fulfilled. The discussion is almost wholly in the form of mathematical equations. It abounds in unfamiliar symbols and in terms such as Hamiltonian operator, secular equations, orthogonality, wave function, overlap integral, linear variation function, and matrix component.

The author's implication that "practically no mathematics" is used in his discussion should not make the American reader feel inferior. No one who is not working in the field or who is not fresh from a study of wave mechanics would be able to follow the mathematical portions of the discussion with understanding without a great deal of digging. Although "mathematics is a language" it is not the native tongue of most chemists, and those who have arrived at useful concepts by means of mathematics and who wish to spread the knowledge of their results must translate them into familiar speech. The argument frequently heard that the only way to express these results precisely is in the form of mathematical equations may be technically correct, but even the mathematician undoubtedly has some picture in mind, because he is quick to point out errors in pictures visualized by the chemist. It is the mathematician's *picture* that most chemists want, and they are quite willing to accept it on faith.

Concerning the content of the book, the molecular-orbital treatment is compared side by side with the valence-bond treatment of diatomic molecules, polyatomic molecules, hybridization, conjugated and aromatic molecules, nonmetallic solids, metals, the hydrogen bond, and hyperconjugation. The conclusion is that both treatments usually arrive at the same result but that the quantitative calculations of certain properties, such as the net charge on an individual atom in a molecule, can be made with less difficulty by the molecular-orbital method. However, most readers would be willing to leave these aspects of the theory of valence to the expert. What is obvious but not emphasized is that the molecular-orbital theory enables the nonmathematically minded reader to visualize the source of bond energy, the nature of directed valence, the cause of restricted rotation about double bonds, the high reactivity of multiple bonds, the nature of conjugation, the conductance of metals, the absorption of light, and many other phenomena more readily than does the valence-bond theory.

Despite the difficulty of wading through the murky mathematical equations, there is much knowledge that chemists can gain by reading this book. The author is careful to point out many misconceptions that have been current as a result, primarily, of attempts by the uninitiated to visualize the valence-bond treatment. The author has a knack for explaining abstruse points, as for example in his discussion of the contribution of atomic dipoles to dipole moments. Largely because there are fewer equations, the last half of the book is easier reading than the first half. A formula index of substances is provided in addition to the author and subject indexes.

CARL R. NOLLER

STANFORD UNIVERSITY
STANFORD, CALIFORNIA

● DANA'S MANUAL OF MINERALOGY

Revised by *Cornelius S. Hurlbut, Jr.,* Associate Professor of Mineralogy, Harvard. Sixteenth edition. John Wiley & Sons, Inc., New York, 1952. viii + 530 pp. 471 figs. 14.5 × 22 cm. $6.

THE several Dana books on mineralogy range from the level of the amateur to that of the professional mineralogist. The "Manual" is pitched at the level of a college student in his first mineralogy course. An indication of its popularity in this use is the fact that it is now in its sixteenth edition. The first edition was by James Dwight Dana in 1848.

The main changes introduced in the present edition are an enlargement of the section on the aims and scope of mineralogy, a change in crystallographic terminology to agree with modern usage, the listing of Hermann-Mauguin symbols for symmetry classes, and a new section on crystal chemistry.

Crystallography is treated from a morphological point of view. Symmetry, axial ratio, parameters, and Miller indexes are explained and the various crystal forms are defined. Physical properties are then discussed. The chapter on chemical mineralogy includes discussions of chemical bonds, atomic packing, polymorphism, and isomorphism, as well as descriptions of simple chemical and blowpipe tests for approximately forty elements.

The main body of the book is the section of descriptions of approximately 200 minerals. Each mineral is described under the headings: crystallography, physical properties, composition, tests, diagnostic features, alteration, occurrence, use, name, and similar species.

Succeeding chapters refer to geologic occurrence and economic uses. There are finally a set of determinative tables, a general index, and a mineral index that lists the main properties.

The book rightly points out that future progress in mineralogy will probably be in the direction of relating properties and occurrences of minerals to their internal structure. The new section on crystal chemistry is a step in this direction. These principles of crystal chemistry are very little applied to specific minerals in this book, however. Presumably the author believes that the first course in mineralogy should aim primarily at training the student in simple methods of identification of the more important minerals. With such training the student can identify minerals in the field for the purpose of making geological interpretations. Later courses, using more advanced textbooks, will deal with such methods of identification as optical properties and X-ray techniques and with the application of crystal chemistry to minerals.

The new edition of Dana's "Manual of Mineralogy" is recommended as a good straightforward textbook for a college introductory course in mineralogy. It stresses identification and also presents basic principles on which further progress can be made.

ALONZO W. QUINN

BROWN UNIVERSITY
PROVIDENCE, RHODE ISLAND

NEWS

BIOCHEMICAL PREPARATIONS SERIES NOW IN HIGH GEAR

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WILEY BOOKS

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. . . *about RICHTER'S* **TEXTBOOK OF ORGANIC CHEMISTRY,** *Third Edition (1952, 762 pages, $6.75).* Donald C. Gregg, in the Journal of Chemical Education: "Any student who digests this text should be a respectable resident of the 'first stage' of proficiency. . . . Teachers of organic chemistry who are considering adoption of a different text for their beginning course might well give this volume serious consideration." Richter's **LABORATORY MANUAL OF ELEMENTARY ORGANIC CHEMISTRY,** *Second Edition (1951, 146 pages, $1.96)* earned this comment by Jacob Sharefkin in the Journal of Chemical Education: ". . . represents a high standard of pedagogical achievement and is enthusiastically recommended by the reviewer."

. . . *about BULL'S* **PHYSICAL BIOCHEMISTRY,** *Second Edition (1951, 255 pages, $5.75).* First of all, keep in mind the important feature of this book—its relentless emphasis on the interrelationship of the physical and biological sciences and on the value of thoroughly understanding purely physical phenomena in studying biological systems. Is it successful? Professor Harold Williams of Cornell University writes: "To me this is an excellent revision and much needed book which I believe will see wider use in the future."

. . . *about LUCAS and PRESSMAN'S* **PRINCIPLES AND PRACTICE IN ORGANIC CHEMISTRY,** *(1949, 557 pages, $6.00).* H. R. Nace, reviewing this book for the Journal of Chemical Education, hit the nail on the head as he wrote: ". . . should prove useful in organic laboratory courses, *especially in view of its flexibility and adaptability to a variety of teaching needs*" (italics ours!). A user comments: "Morale has seemed much better, for the students seemed to take a greater-than-usual interest in the experiments they were doing. I have liked to teach from it and am particularly pleased with the adequate discussions of collateral material." Professors and instructors who want a manual that shows the student how to use his *head* as well as his hands might examine this book for possible use in their courses.

. . . and a reminder about the **METHUEN MONOGRAPHS IN CHEMICAL AND BIOCHEMICAL SUBJECTS,** now distributed in the U. S. exclusively by Wiley. These little books have met with considerable success as reviews or introductions to fields which are on the borderline of the reader's main interest. **SELECTIVE TOXICITY WITH SPECIAL REFERENCE TO CHEMOTHERAPY,** *by A. ALBERT (228 pages, $1.75)* . . . **BIOCHEMISTRY OF THE NUCLEIC ACIDS** *by J. N. DAVIDSON (163 pages, $1.75)* . . . **ACIDS AND BASES,** *by R. P. BELL (90 pages, $1.50)* . . . **THE THERMODYNAMICS OF THE STEADY STATE,** *by K. G. DENBIGH (103 pages $1.75)* . . . **FLUORINE AND ITS COMPOUNDS,** *by R. N. HASZELDINE and A. G. SHARPE (153 pages, $1.75).*

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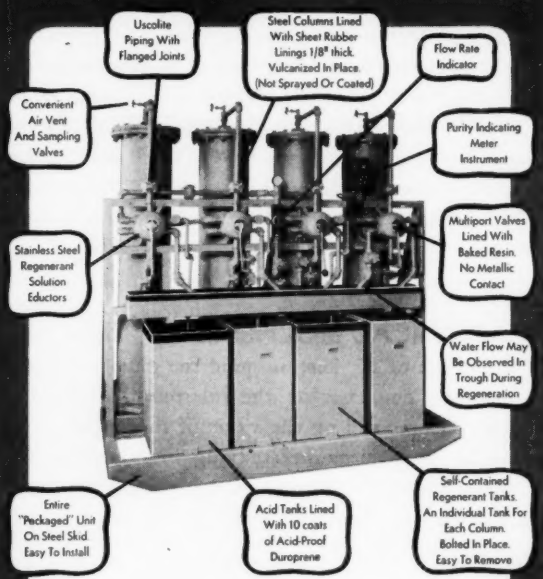
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Out of the Editor's Basket

New Universal Balance

A completely new instrument, the Voland Universal Balance, which is said to combine the speed of the fastest projection balance with an accuracy that exceeds the standards required of the finest analytical balance, has recently been introduced by Voland & Sons, Inc., 32 Relyea Place, New Rochelle, New York.

In effect, two balances in one, the Voland Universal is first, a high speed projection balance with a superior stability reading to 0.1 milligram. In addition, it can be used as an analytical balance with readings to 0.01 milligram by means of a simple sensitivity shift.

Weighings to an accurate fourth decimal place are obtained in seconds by straight projection, or to the fifth decimal place by shifting sensitivity and adding an additional decade of weights.

All weights from 0.01 to 200 grams are built in the balance and are obtained by manipulating five selector knobs. Speed is gained by projection reading, eliminating need for weights in lower ranges. Weights can be readily lifted out of their supporting members for recalibration or cleaning.

Complete specifications on the Voland Universal Balance are available from the manufacturer.

Effects of Sound Energy

New fields of research into the effect of sound energy on organic and inorganic materials have recently been opened up with the development of the magnetostriction oscillator by the Raytheon Manufacturing Co. of Waltham, Massachusetts.

With this inexpensive but powerful unit almost any laboratory can conduct its own studies into the effects of sound energy on liquids, living organisms, mixtures, and chemical reactions.

Successful applications include: acceleration of bacteria growth, disintegration of bacteria for antigen extraction, acceleration of seed germination, dispersion of antibiotic crystals for infection purposes, diffusion of gases from liquids, acceleration of chemical reactions, decomposition of gelatins and gums, emulsification of liquids, and dispersion of agglomerations in liquids.

The oscillator is composed of three parts: a driving element, a stand containing a drive coil, and a rod-and-cup assembly. The unit derives its power when the rod which consists of a series of nickel laminations, is subjected to an alternating magnetic field. This causes it to oscillate longitudinally, vibrating the diaphragm which forms the bottom of the cup. The cup itself is water-cooled to allow testing under close temperature control. During the vibrational treatment, tested liquids contact only stainless materials.

Valves

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Among subjects in the Spring '52 issue are:

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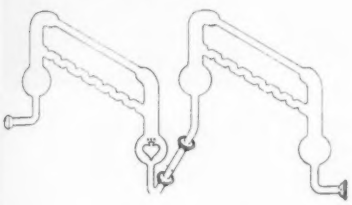
Ace Glass, Inc., Vineland, New Jersey, announces a new type gas washing apparatus which is said to offer numerous advantages over the conventional type bottle and stopper arrangement.



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New Bibliography of Applications for Spectrophotometers

A helpful new Bibliography of Applications for the Beckman Models B and DU Spectrophotometers is now available. More than 500 specific sources of application data for these well-known analytical instruments are listed. The first section is devoted to identification, analysis, and color measurement and embraces such fields as: General Organic and Inorganic Chemistry; Metals; Petroleum Derivatives and Fuels; Glass, Plastics, Resins, and Rubber; Biologicals, Medicine, Pharmaceuticals; Vitamins; Foods and Beverages; Oils, Fatty Acids, and Pigments; Cosmetics, Clays, Paints, and Papers; Textiles, Dyes, and Leather; Plant Materials, Soils, and Insecticides.

Special Sections provide up-to-the-minute data on Structural Identification, Rate and Equilibria Studies, Emission (Including Flame Spectrophotometry), Special Instrumentation and Standards, Calibration, Filters, Theory and Miscellaneous References.

This new edition represents the very latest source material for users or those interested in spectrophotometric methods. Industrial as well as laboratory problems are represented. Copies of this new publication may be obtained by addressing request to Beckman Instruments, Inc., South Pasadena, California. Ask for Bulletin 36-222.

New Literature

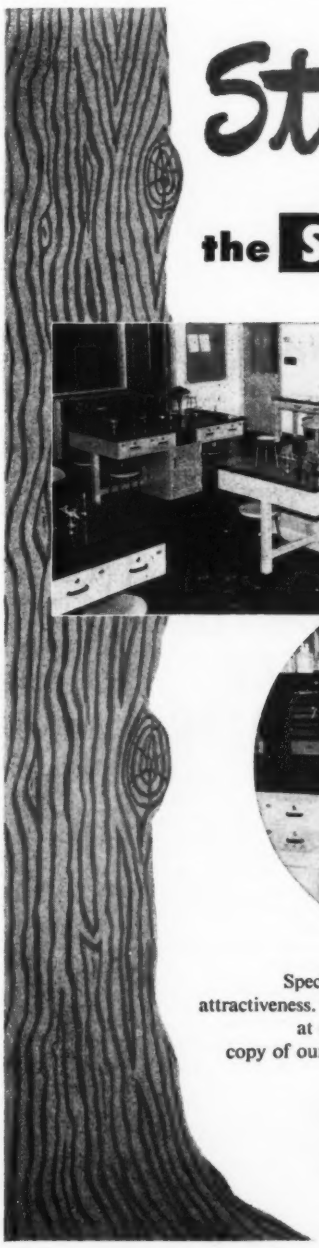
Performance and construction of the Model 21 Double Beam Recording Infrared Spectrophotometer are described in Bulletin 104, an illustrated 36-page brochure published by The Perkin-Elmer Corp., Norwalk, Connecticut.

Included in the new bulletin are background sections on infrared spectroscopy and the optical null principle employed in the Model 21. Complete descriptions of the optical, detector and amplifier systems, as well as performance data and accessories, are also included.

Bulletin 104 is the latest in an illustrated series describing Perkin-Elmer instruments. It is available from The Perkin-Elmer Corp., Norwalk, Connecticut.

An attractive bulletin just off the press is offered with the latest information on the Heavy Duty Chem-O-Feeder. These small proportioning pumps, available with one, two, or three measuring chambers for capacities up to 57 gph., are supplied as a "package," making installation extremely simple. The bulletin gives specifications and features together with many installation diagrams, a list of chemicals fed, and other helpful information. For copies of Bulletin No. 1225 write the manufacturer, %Proportioneers, Inc.%, Providence 1, Rhode Island.

An attractive, four-page bulletin illustrating and describing the new Metrogram balance has just been published by Arthur S. LaPine and Co. Copies may be obtained by writing to 6001 S. Knox Avenue, Chicago 29, Illinois.



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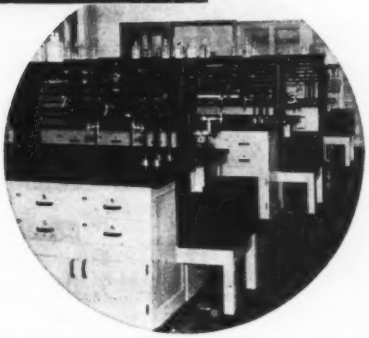

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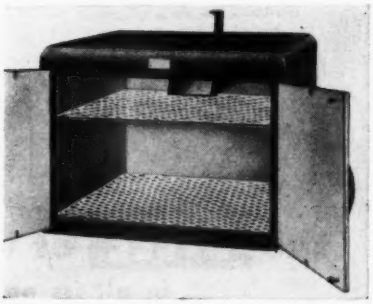

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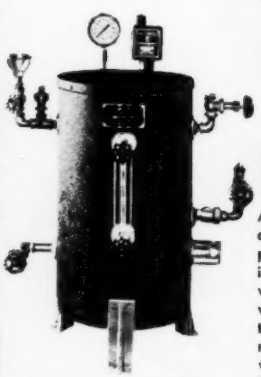

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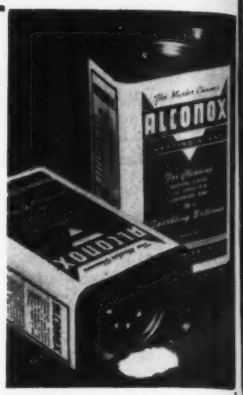

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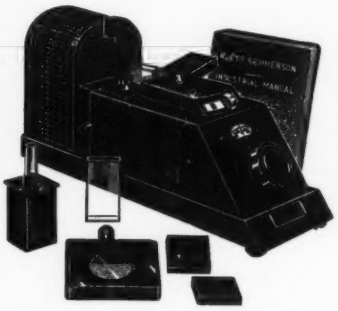

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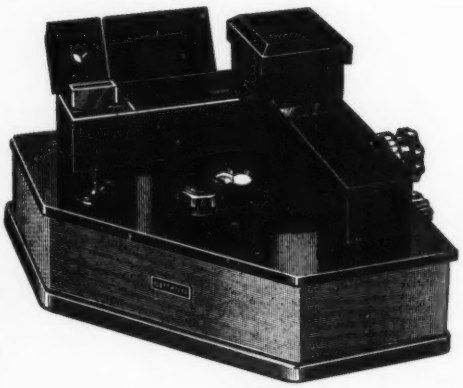

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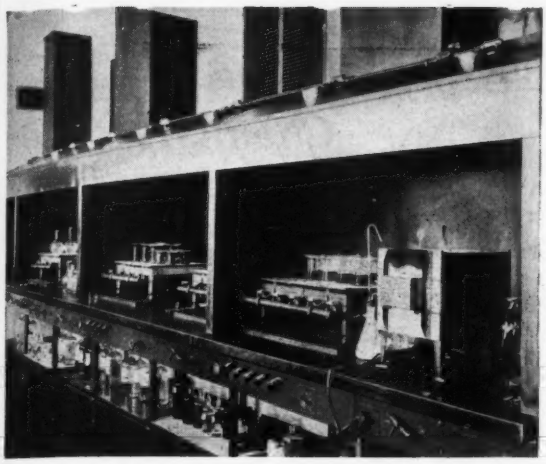

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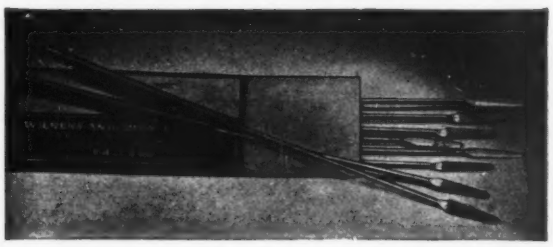

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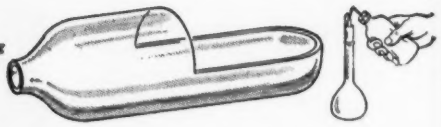

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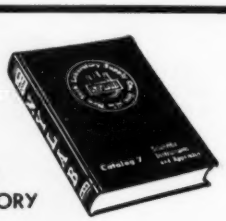

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References

(1) Ind. Eng. Chem. 25-653 (June, 1933)

(2) Ind. Eng. Chem. 25-1112 (Oct., 1933)

(3) National Bureau of Standards Journal of Research 12-241 (Feb. 1934, R.P. No. 649)

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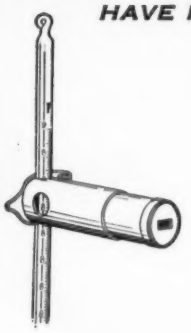

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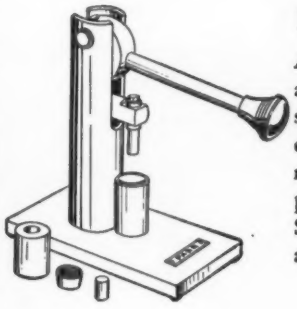

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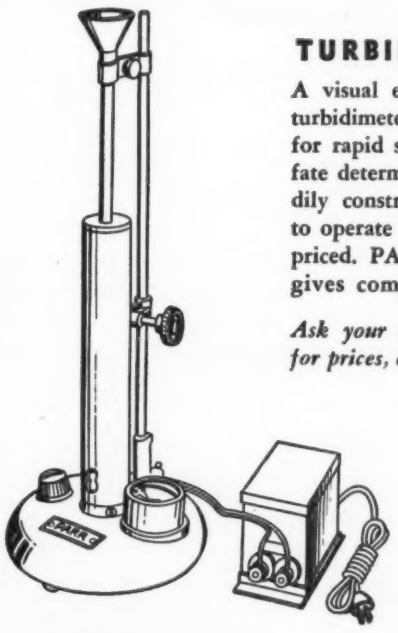

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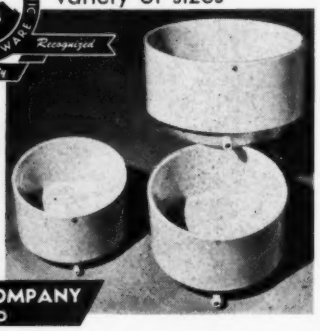

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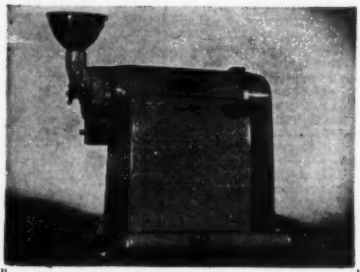

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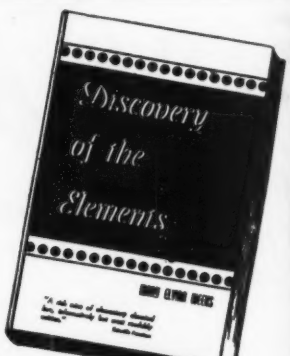

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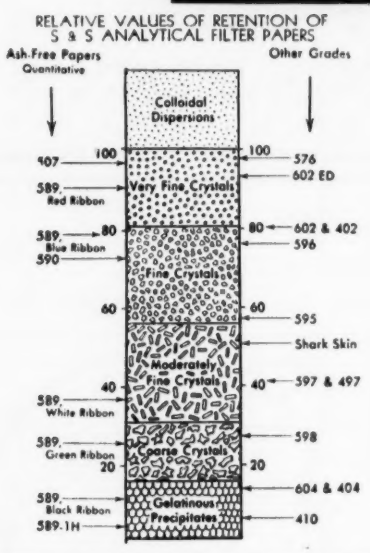

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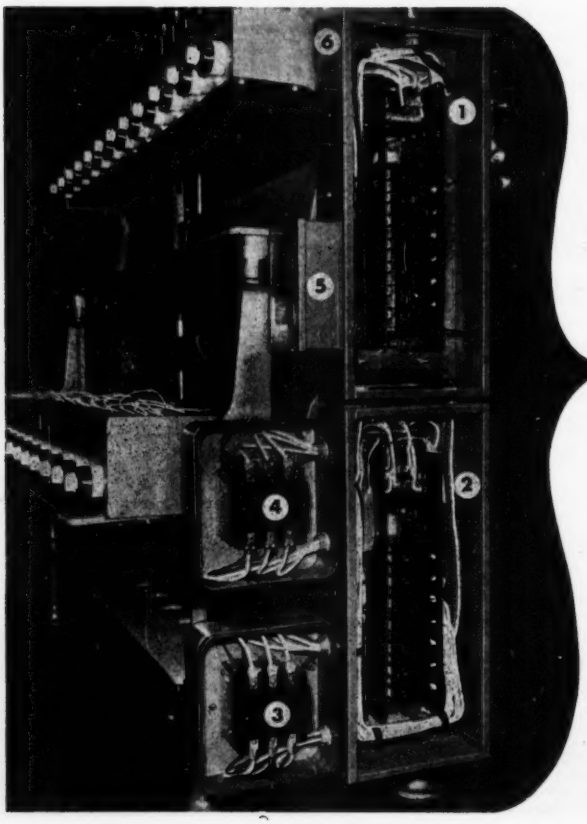

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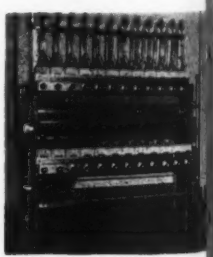

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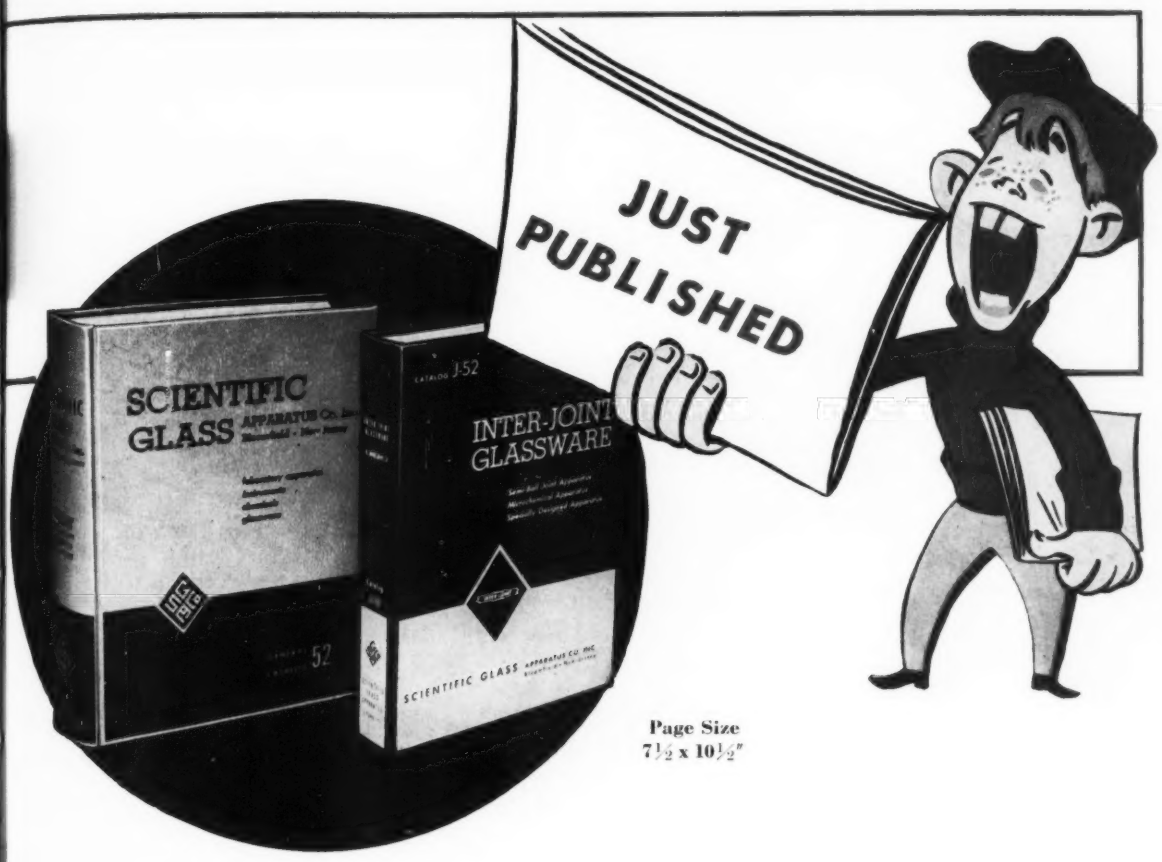

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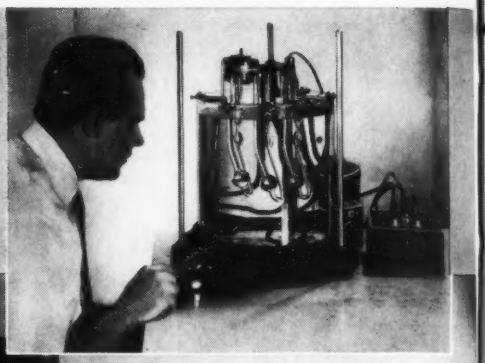

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